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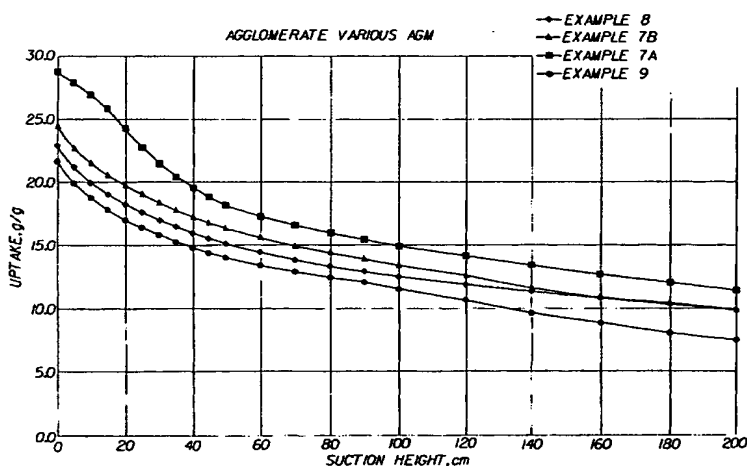
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(54) Title: PRODUCT WITH ABSORBENT POLYMER AND PARTICULATE FOAM



(57) Abstract: The present invention is a high capillary suction storage absorbent member comprising an agglomerate of particulate hydrogel-forming absorbent polymer and particulate high surface area open-celled hydrophilic foam. In another aspect of the invention the agglomerate of particulate hydrogel-forming absorbent polymer and particulate high surface area open-celled hydrophilic foam is disclosed. The absorbent member is useful in the containment (e.g., storage) of body liquids such as urine. In one embodiment, the storage absorbent member comprises the agglomerate. In another embodiment, the absorbent member comprises the agglomerate adjacent to at least one sheet, strip or piece of high surface area open-celled hydrophilic foam material. In either the storage absorbent member or agglomerates of the invention, the high surface area open-celled hydrophilic foam comprises from about 1 % to about 98 %, preferably from about 15 % to about 85 %, more preferably from about 30 % to about 40 % by weight of the member or agglomerate, respectively. An unique aspect of the agglomerates according to the present invention is that they are formed without the use of a separate bonding substance, such as an adhesive.

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## PRODUCT WITH ABSORBENT POLYMER AND PARTICULATE FOAM

CROSS-REFERENCE TO RELATED APPLICATIONS

5 This application is a continuation-in-part of co-pending U.S. Application Serial No. 09/258,889, filed March 1, 1999, which is a continuation-in-part of U.S. Application Serial No. 09/041,930, filed March 13, 1998, which is a continuation-in-part of U.S. Application Serial No. 08/721,648, filed September 26, 1996 (now U.S. Patent 5,744,506), which is a divisional of U.S. Application Serial No. 08/655,041, filed  
10 May 28, 1996 (now U.S. Patent 5,741,581), which is a divisional of U.S. Application Serial No. 08/563,866, filed November 29, 1995 (now U.S. Patent 5,650,222), which is a continuation of U.S. Application Serial No. 08/370,922, filed January 10, 1995 (now abandoned). Applicants claim benefit to these applications pursuant to 35 U.S.C. §120.

FIELD OF THE INVENTION

15 This application relates to a high capillary suction storage absorbent member that is particularly suitable for absorbing aqueous bodily fluids such as urine.

BACKGROUND OF THE INVENTION

The development of highly absorbent members for use as disposable diapers, adult incontinence pads and briefs, and catamenial products such as sanitary napkins,  
20 are the subject of substantial commercial interest. A highly desired characteristic for such products is thinness. For example, thinner diapers are less bulky to wear, fit better under clothing, and are less noticeable. They are also more compact in the package, making the diapers easier for the consumer to carry and store. Compactness in packaging also results in reduced distribution costs for the manufacturer and distributor,  
25 including less shelf space required in the store per diaper unit.

The ability to provide thinner absorbent articles such as diapers has been contingent on the ability to develop relatively thin absorbent cores or structures that can acquire and store large quantities of discharged body liquids, in particular urine. In this regard, the use of certain absorbent polymers often referred to as "hydrogels,"  
30 "superabsorbents" or "hydrocolloid" material has been particularly important. See, for example, U.S. Patent 3,669,103 (Harper et al.), issued June 13, 1972, and U.S. Patent 3,670,731 (Harmon), issued June 20, 1972, that disclose the use of such absorbent polymers (hereafter referred to as "hydrogel-forming absorbent polymers") in absorbent articles. Indeed, the development of thinner diapers has been the direct consequence of

thinner absorbent cores that take advantage of the ability of these hydrogel-forming absorbent polymers to absorb large quantities of discharged body liquids, typically when used in combination with a fibrous matrix. See, for example, U.S. Patent 4,673,402 (Weisman et al.), issued June 16, 1987 and U.S. Patent 4,935,022 (Lash et al.), issued June 19, 1990, that disclose dual-layer core structures comprising a fibrous matrix and hydrogel-forming absorbent polymers useful in fashioning thin, compact, nonbulky diapers. See also, U.S. Patent 5,562,646 (Goldman et al.), issued Oct. 8, 1996 and U.S. Patent 5,599,335 (Goldman et al.), issued Feb. 4, 1997, both of which relate to absorbent cores comprising regions of high concentrations of hydrogel-forming absorbent polymer, where the absorbent polymer forms a gel-continuous liquid transportation zone upon swelling.

In addition to the use of hydrogel-forming absorbent polymers as the primary component in absorbent article storage structures, the use of high surface area open-celled hydrophilic foam materials derived from high internal phase water-in-oil emulsions ("HIPEs") has been identified. See, e.g., U.S. Patent No. 5,260,345 (DesMarais et al.), issued November 9, 1993, U.S. Patent No. 5,387,207 (Dyer et al.) issued Feb. 7, 1995, and U.S. Patent No. 5,560,222 (DesMarais et al.), issued July 22, 1997. The foam materials, particularly those designed to function as liquid storage/redistribution components, provide several advantages over storage structures comprising hydrogel-forming absorbent polymers in a fibrous matrix, including good wicking and liquid distribution characteristics, high storage capacity under pressure, flexibility, etc.

The primary focus of prior work in both the hydrogel-forming absorbent polymer and HIPE foam areas has been the maximization of liquid storage capacity in a relatively thin material. Hydrogel-forming absorbent polymer materials absorb liquid and provide leakage protection and dryness in absorbent products. Once absorbed, the liquid in absorbent polymers is tightly held by osmotic forces, which helps prevent rewet of the topsheet by previously absorbed urine. However, hydrogel-forming polymer by itself has little ability to absorb liquid if the liquid is not delivered to its surface. This is especially critical at high capillary heights where the liquid is present only in small capillaries. For example, conventional softwood pulp exhibits almost no uptake at capillary suction heights of 100 cm. It is not surprising, then, that a mixture of pulp and hydrogel-forming polymer exhibits almost no uptake at 100 cm. Thus, in spite of the advancements made to achieve the goal of high liquid storage capacity in thin materials, there is a continuing need to provide high storage capacity materials that also exhibit high capillary suction

capabilities. Storage materials which exhibit high capillary suction capacity will allow the dewatering of other absorbent core materials such as acquisition and distribution materials, one or both of which are typically included in absorbent cores of absorbent articles. By thoroughly dewatering these other absorbent core components, those materials will be better able to handle additional insults of liquid by the wearer. In addition to high capillary suction capacities in general, a particularly desirable property is the ability to provide such capacities at relatively high capillary suction heights. Movement of liquid from the discharge region (i.e., the crotch region of the article) to the front and rear of the article may provide enhanced wearer comfort when the article is wetted with liquid. As is clear, the ability of a storage material to dewater other core components, particularly the distribution material that wicks liquid to high capillary heights, is particularly relevant to their functioning as absorbent materials in absorbent articles.

Accordingly, it would be beneficial to provide a storage absorbent member having a high liquid storage capacity along with a high capillary suction capability. Ease of manufacture and reduced manufacturing costs are additional beneficial properties.

#### SUMMARY OF THE INVENTION

The present invention is a high capillary suction storage absorbent member comprising an agglomerate of particulate hydrogel-forming absorbent polymer and particulate high surface area open-celled hydrophilic foam. In another aspect of the invention the agglomerate of particulate hydrogel-forming absorbent polymer and particulate high surface area open-celled hydrophilic foam is disclosed. The absorbent member is useful in the containment (e.g., storage) of body liquids such as urine. In one embodiment the absorbent member comprises the agglomerate. In another embodiment, the absorbent member comprises the agglomerate adjacent to at least one sheet, strip or piece of high surface area open-celled hydrophilic foam material.

### DETAILED DESCRIPTION OF THE DRAWINGS

Figure 1 is a graph showing the capillary sorption isotherms for various agglomerates of the present invention.

Figure 2 is a photomicrograph (100X magnification) of an agglomerate of the present invention wherein numerous particles of high surface area open-celled hydrophilic foam are bonded to a particle of a hydrogel-forming absorbent polymer. A is defining the area of an agglomerate which is magnified by 800X in Figure 3. B is illustrating particles of high surface area open-celled hydrophilic foam. C is illustrating particles of high surface area open-celled hydrophilic foam.

Figure 3 is a photomicrograph of the agglomerate of Figure 2 at increased magnification (800 X).

Figure 4 of the drawings is an exploded view of a diaper having an absorbent core which comprises a high capillary suction capacity storage absorbent member of the present invention.

Figure 5A is a schematic view of an apparatus for measuring capillary sorption absorbent capacity of an absorbent member.

Figure 5B is a cross-sectional, close-up view of the glass frit shown generally in Figure 5A.

Figure 5C is a cross-sectional, close-up view of the cylinder/piston assembly of the glass frit shown in Figure 5B.

Figure 5D is a cross-sectional, close-up view of the piston aspect of the cylinder/piston assembly shown in Figure 5C.

Figure 6 is an illustration of storage absorbent members of the invention. Figure 6(a) illustrates a structure comprising an agglomerate of particulate hydrogel-forming absorbent polymer and particulate high surface area open-celled hydrophilic foam positioned between two sheets of high surface area open-celled hydrophilic foam material. Figure 6(b) illustrates a structure comprising an agglomerate of particulate hydrogel-forming absorbent polymer and particulate high surface area open-celled hydrophilic foam adjacent one sheet of high surface area open-celled hydrophilic foam material. Figure 6(c) illustrates a structure comprising multiple layers of an agglomerate of particulate hydrogel-forming absorbent polymer and particulate high surface area open-celled hydrophilic foam adjacent multiple sheets of high surface area open-celled hydrophilic foam material.



## DETAILED DESCRIPTION OF THE INVENTION

### I. Definitions

As used herein, the term "body liquids" includes, but is not limited to, urine, menses, vaginal discharges, sweat and feces.

5 As used herein, the term "agglomerate" refers to a particulate unitary combination of particulate hydrogel-forming absorbent polymer and particulate high surface area open-celled hydrophilic foam that is not easily separable. By not easily separable is preferably meant, a combination that does not substantially separate into its component particles (i.e., hydrogel-forming absorbent polymer particles and high surface area open-celled foam particles) as a result of normal manufacturing, normal shipping and/or  
10 normal use.

As used herein, the term "absorbent core" refers to the component of the absorbent article that is primarily responsible for liquid handling properties of the article, including acquiring, transporting, distributing and storing body liquids. As such, the  
15 absorbent core typically does not include the topsheet or backsheet of the absorbent article.

As used herein, the term "absorbent member" refers to the components of the absorbent core that typically provide one or more liquid handling properties, e.g., liquid acquisition, liquid distribution, liquid transportation, liquid storage, etc. The absorbent  
20 member can comprise the entire absorbent core or only a portion of the absorbent core, i.e., the absorbent core can comprise one or more absorbent members. The "storage absorbent member" is the absorbent member component(s) of the absorbent core that functions primarily to store absorbed liquids. As discussed above, the storage absorbent member may also function to distribute liquid as a result of its vertical wicking capability.

25 As use herein, the term "layer" refers to an absorbent member whose primary dimension is X-Y, i.e., along its length and width. It should be understood that the term layer is not necessarily limited to single layers or sheets of material. Thus the layer can comprise laminates or combinations of several sheets or webs of the requisite type of materials. Accordingly, the term "layer" includes the terms "layers" and "layered."

30 As used herein, the term "osmotic absorbent" refers to a material or structure that absorbs solution in response to a chemical potential difference between absorbed and non-absorbed solutions. Generally, this chemical potential difference arises from a higher solute concentration for the absorbed solution. In order to inhibit equalization of solute concentration via diffusion of solute species, an osmotic absorbent typically has a

diffusion barrier that selectively inhibits the diffusion of at least one solute species. Examples of suitable diffusion barriers are (i) a semi-permeable reverse-osmosis membrane, wherein the membrane provides a diffusion barrier to soluble salts (e.g., NaCl) and (ii) a crosslinked polyelectrolyte network (e.g., used in hydrogels), wherein the polyelectrolyte network retains dissociated counterions inside the gel as a result of electroneutrality considerations. Examples of osmotic packet or chamber absorbents are described in U.S. Patent No. 5,108,383 issued April 28, 1992 to White and U.S. Patent No. 5,082,723 issued Jan. 21, 1992 to Gross et al., the disclosure of each of which is incorporated herein by reference. The osmotic absorbent for use in the storage absorbent members and agglomerates of the present invention are hydrogel-forming absorbent polymers, which are described in detail below.

As used herein, the term "X-Y dimension" refers to the plane orthogonal to the thickness of the member, core or article. The X-Y dimension usually corresponds to the length and width, respectively, of the member, core or article.

As used herein, the terms "region(s)" or "zone(s)" refer to portions or sections of the absorbent member.

As used herein, the term "Z-dimension" refers to the dimension orthogonal to the length and width of the member, core or article. The Z-dimension usually corresponds to the thickness of the member, core or article.

For purposes of this invention, it should also be understood that the term "upper" refers to absorbent members, such as layers, that are nearest to the wearer of the absorbent article, and typically are relatively proximate the topsheet of an absorbent article; conversely, the term "lower" refers to absorbent members that are furthestmost away from the wearer of the absorbent article and typically are more proximate the backsheet.

As used herein, the term "comprising" means various components, members, steps and the like can be conjointly employed according to the present invention. Accordingly, the term "comprising" encompasses the more restrictive terms "consisting essentially of" and "consisting of," these latter, more restrictive terms having their standard meaning as understood in the art.

All percentages, ratios and proportions used herein are by weight unless otherwise specified.

II. Capillary Suction Properties of Storage Absorbent Members of the Invention and of the Agglomerates Utilized Therein

The agglomerates utilized in the storage absorbent members of the present invention exhibit high capillary suction capacities. Accordingly, when such agglomerates are incorporated into storage absorbent members, the storage absorbent members will also exhibit high capillary suction capacities. The properties exhibited by the storage absorbent members (capillary suction capacity, as well as other properties discussed below) will be dependent upon the corresponding properties of the agglomerate incorporated into the absorbent member; the degree to which the properties of the agglomerate and storage absorbent member differ will be dependent upon the type and amount of any additional materials incorporated into the storage absorbent member. Accordingly, while many properties are discussed herein with reference to an agglomerate, it should be understood that a storage absorbent member comprising such an agglomerate may exhibit similar properties and therefore, the properties described and ranges provided apply equally to storage absorbent members of the present invention.

The high suction capacity is measured in terms of the agglomerate's ability to uptake liquid at high capillary heights, which are generally encountered when the member is positioned in an absorbent article. The Capillary Sorption Absorbent Capacity test (also referred to herein as the Capillary Sorption test) measures the amount of test liquid per gram of agglomerate that is taken up when the agglomerate is placed at varying heights on a capillary sorption apparatus. The Capillary Sorption Absorbent Capacity test is described in greater detail in the Test Methods section below. Capillary sorption isotherms for representative agglomerates are depicted graphically in Figure 1. In particular, capillary sorption isotherms are depicted for (i) an agglomerate made in accordance with Example 8, herein, where the particulate hydrogel-forming absorbent polymer is ASAP 2300 (available from Chemdal Corporation of Palantine, IL; also available from The Procter & Gamble Co., Paper Technology Division, Cincinnati, OH) and the particulate open-celled hydrophilic foam is made in accordance with Example 4; (ii) an agglomerate made in accordance with Example 7B herein, where the particulate hydrogel-forming absorbent polymer is a homogeneously crosslinked polymer (as described in Example 6 herein) and the particulate high surface area open-celled hydrophilic foam is made in accordance with Example 4 herein; (iii) an agglomerate

made in accordance with Example 7A herein, where the particulate hydrogel-forming absorbent polymer is a mixed bed ion exchange ("MBIE") absorbent polymer (as described in Example 5 herein) and the particulate high surface area open-celled hydrophilic foam is made in accordance with Example 4 herein ; and (iv) an agglomerate  
5 made in accordance with Example 9 herein, where the particulate hydrogel-forming absorbent polymer is Favor 1180 (available from Stockhausen Louisiana LLC, also available from The Procter & Gamble Co., Paper Technology Division, Cincinnati, OH) and the particulate high surface area open-celled hydrophilic foam is made in accordance with Example 4 herein.

10 In one aspect, the agglomerate of the present invention has a capillary sorption absorbent capacity at a height of 35 cm of at least about 12 g/g, preferably at least about 14 g/g, more preferably at least about 16 g/g, more preferably at least about 20 g/g, more preferably at least about 22 g/g, still more preferably at least about 35 g/g. Typically, these agglomerates will have a capillary sorption absorbent capacity at a  
15 height of 35 cm of from about 12 g/g to about 60 g/g or to as much as about 70 g/g, more typically from about 14 g/g to about 50 g/g or to as much as about 60 g/g, more typically from about 16 g/g to about 40 g/g or to as much as about 55 g/g, more typically from about 20 g/g to about 50 g/g, more typically from about 22 g/g to about 45 g/g.

In another aspect, the agglomerate has a capillary sorption absorbent capacity at  
20 a height of 50 cm of at least about 7 g/g, preferably at least about 9 g/g, more preferably at least about 12 g/g, still more preferably at least about 16 g/g, still more preferably at least about 21 g/g, and still more preferably at least about 30 g/g. Typically, these agglomerates will have a capillary sorption absorbent capacity at a height of 50 cm of from about 7 g/g to about 40 g/g or to as much as about 60 g/g, more typically from  
25 about 9 g/g to about 35 g/g or to as much as about 50 g/g, still more typically from about 12 g/g to about 30 g/g or to as much as about 45 g/g, still more typically from about 16 g/g to about 40 g/g, and still more typically from about 21 g/g to about 35 g/g.

In yet another aspect, the agglomerate has a capillary sorption absorbent capacity at a height of 100 cm of at least about 4 g/g, preferably at least about 6 g/g,  
30 more preferably at least about 8 g/g, still more preferably at least about 12 g/g, still more preferably at least about 17 g/g, and still more preferably at least about 25 g/g. Typically, these agglomerates will have a capillary sorption absorbent capacity at a height of 100 cm of from about 4 g/g to about 30 or to as much as about 50 g/g, more typically from about 6 g/g to about 25 g/g or to as much as about 45 g/g, still more typically from about

8 g/g to about 20 g/g or to as much as about 40 g/g, still more typically from about 12 g/g to about 35 g/g, and still more typically from about 17 g/g to about 30 g/g.

5 In yet another aspect, the agglomerate has a capillary sorption absorbent capacity at a height of 140 cm of at least about 4 g/g, preferably at least about 5 g/g, more preferably at least about 7 g/g, still more preferably at least about 10 g/g, still more preferably at least about 14 g/g, and still more preferably at least about 23 g/g. Typically, these agglomerates will have a capillary sorption absorbent capacity at a height of 140 cm of from about 4 g/g to about 28 g/g or to as much as about 45 g/g, more typically from about 5 g/g to about 23 g/g or to as much as about 40 g/g, still more typically from about 7 g/g to about 18 g/g or to as much as about 35 g/g, still more typically from about 10 g/g to about 30 g/g, and still more typically from about 14 g/g to about 25 g/g.

10 In yet another aspect, the agglomerate has a capillary sorption absorbent capacity at a height of 200 cm of at least about 3 g/g, preferably at least about 4 g/g, more preferably at least about 6 g/g, still more preferably at least about 8 g/g, still more preferably at least about 11 g/g, and still more preferably at least about 20 g/g. Typically, these agglomerates will have a capillary sorption absorbent capacity at a height of 200 cm of from about 3 g/g to about 25 g/g or to as much as about 40 g/g, more typically from about 4 g/g to about 20 g/g or to as much as about 35 g/g, still more typically from about 6 g/g to about 15 g/g or to as much as about 30 g/g, still more typically from about 8 g/g to about 25 g/g, and still more typically from about 11 g/g to about 23 g/g.

20 In addition, or alternative, to defining the high capillary suction capabilities of the agglomerates in terms of capillary sorption absorbent capacity, the high capillary suction capabilities may be characterized by the agglomerate's ability to initially uptake liquid at high heights at relatively fast rates. High capillary suction agglomerates that exhibit both high uptake at high suction and high initial effective uptake rates should provide superior user dryness as the extent of partitioning from other absorbent storage member or absorbent core member materials (e.g., acquisition or distribution materials) and its rate will be favorably improved by the high capillary suction material. For purposes of the present disclosure, this latter property is referred to herein as the member's "initial effective uptake rate at 200 cm capillary suction height" (referred to herein as "initial effective uptake rate at 200 cm"), which is reported in units of g/g/hour. The initial effective uptake rate of an agglomerate is calculated by dividing the capillary suction absorbent capacity at 200 cm by the time spent at 200 cm. Capillary suction absorbent capacity and time are readily determined using the Capillary Sorption method discussed

in detail in the Test Methods section below. Though not a requirement, particularly preferred agglomerates will have an initial effective uptake rate at 200 cm of at least about 3 g/g/hr, more preferably at least about 4 g/g/hr, and most preferably at least about 8 g/g/hr. Typically, the effective uptake rate at 200 cm will be from about 3 to about 15 g/g/hr, more typically from about 4 to about 12 g/g/hr, still more typically from about 8 to about 12 g/g/hr.

While the above minimum capillary sorption absorbent capacities are important to the agglomerates of the present invention, the agglomerates will also preferably, though not necessarily, have a capillary sorption absorbent capacity at zero head pressure (i.e., at 0 cm in the Capillary Sorption test) of at least about 15 g/g. In another preferred aspect, the agglomerates concurrently exhibit the required g/g uptake at least two suction heights discussed above. That is, for example, preferred agglomerates will have 2 or more of the following properties: (i) a capillary sorption absorbent capacity at a height of 35 cm of at least about 12 g/g, preferably at least about 14 g/g, more preferably at least about 16 g/g, still more preferably at least about 20 g/g, still more preferably at least about 22 g/g, and still more preferably at least about 35 g/g; (ii) a capillary sorption absorbent capacity at a height of 50 cm of at least about 7 g/g, preferably at least about 9 g/g, more preferably at least about 12 g/g, still more preferably at least about 16 g/g, still more preferably at least about 21 g/g, and still more preferably at least about 30 g/g; (iii) a capillary sorption absorbent capacity at a height of 100 cm of at least about 4 g/g, preferably at least about 6 g/g, more preferably at least about 8 g/g, still more preferably at least about 12 g/g, still more preferably at least about 17 g/g, and still more preferably at least about 25 g/g; (iv) a capillary sorption absorbent capacity at a height of 140 cm of at least about 4 g/g, preferably at least about 5 g/g, more preferably at least about 7 g/g, still more preferably at least about 10 g/g, still more preferably at least about 14 g/g, and still more preferably at least about 23 g/g; (v) a capillary sorption absorbent capacity at a height of 200 cm of at least about 3 g/g, preferably at least about 4 g/g, more preferably at least about 6 g/g, still more preferably at least about 8 g/g, still more preferably at least about 11 g/g, and still more preferably at least about 20 g/g.

In yet another aspect, agglomerates of the present invention can be characterized in terms of exhibiting a relatively high absorbency efficiency (hereafter referred to as "capillary absorption efficiency") at various heights, relative to the material's capacity at zero head pressure. Capillary absorption efficiency at a given suction height is determined by dividing the capillary suction absorbent capacity of the

material at that given height by the capillary suction absorbent capacity of that material at zero head pressure. In this regard, in one aspect, the agglomerate will have a capillary sorption absorbent capacity at zero height of at least about 15 g/g, preferably at least about 20 g/g, more preferably at least about 40 g/g and most preferably about 60 g/g, and capillary absorption efficiency at a height of 100 cm of at least about 25%, preferably at least about 40%, still more preferably at least about 60%, and still more preferably at least about 70%. In another aspect, the agglomerate will have a capillary sorption absorbent capacity at zero height of at least about 15 g/g, preferably at least about 20 g/g, more preferably at least about 40 g/g and most preferably at least about 60 g/g, and a capillary absorption efficiency at a height of 50 cm of at least about 30%, preferably at least about 50%, still more preferably at least about 70%, and still more preferably at least about 80%. In still another aspect, the agglomerate will have a capillary sorption absorbent capacity at zero height of at least about 15 g/g, preferably about 20g/g, more preferably about 40 g/g and most preferably about 60 g/g, and a capillary absorption efficiency at a height of 35 cm of at least about 50%, preferably at least about 70%, still more preferably at least about 85%, and still more preferably at least about 90%.

In another aspect, preferred agglomerates of the present invention will have a relatively high medium absorption height, which is defined as the height at which the agglomerate has a capillary sorption absorbent capacity that is 50% of the capillary sorption absorbent capacity at 0 cm height. In this regard, preferred agglomerates will have a capillary sorption absorbent capacity at zero height of at least about 15 g/g, preferably at least about 20 g/g, more preferably at least about 40 g/g and most preferably about 60 g/g, and a medium absorption height of at least about 35 cm, more preferably at least about 40 cm, still more preferably at least about 50 cm, still more preferably at least about 60 cm, still more preferably at least about 100 cm, still more preferably at least about 130 cm, and most still more preferably at least about 200 cm.

### III. Components of the High Suction Storage Absorbent Members

As indicated above, the storage absorbent members of the present invention comprise an agglomerate formed of particulate hydrogel-forming absorbent polymer and a particulate high surface area open-celled hydrophilic foam that facilitates transport of body fluids to the hydrogel-forming absorbent polymer. The storage absorbent member may alternatively comprise an agglomerate adjacent to at least one sheet, strip or piece of high surface area open-celled hydrophilic foam material; for example, as shown schematically in Figure 6. In general, incorporating the particulate hydrogel-forming

absorbent polymer and particulate high surface area open-celled hydrophilic foam into an agglomerate makes construction of a storage absorbent member easier, and makes general handling of the respective particulates easier. Because the particles of hydrogel-forming absorbent polymer have a much higher density relative to the particles of high surface area open-celled hydrophilic foam, the polymer particles have a tendency to sift or settle out of a mixture of the two unless incorporated into an agglomerate, or some similar structure. Representative materials useful in preparing agglomerates and storage absorbent members of the present invention are described in detail below.

A. Hydrogel-Forming Absorbent Polymers

1. Chemical Composition

The agglomerates of the present invention comprise at least one particle of hydrogel-forming absorbent polymer (also referred to as hydrogel-forming polymer). The agglomerates of the present invention may be formed from particles of one type of hydrogel-forming absorbent polymer or from particles of more than one type of hydrogel-forming absorbent polymer. The hydrogel-forming absorbent polymer is an osmotic absorbent. Hydrogel-forming polymers useful in the present invention include a variety of water-insoluble, but water-swellaable polymers capable of absorbing large quantities of liquids. Such hydrogel-forming polymers are well known in the art and any of these materials are useful in the high capillary suction storage absorbent members and agglomerates of the present invention. An example of a hydrogel-forming absorbent polymer suitable for use in the invention is ASAP 2300, available from Chemdal Corporation of Palantine, IL; also available from The Procter & Gamble Co., Paper Technology Division, Cincinnati, OH.

Hydrogel-forming absorbent polymers materials are also commonly referred to as "hydrocolloids," or "superabsorbent" materials and can include polysaccharides such as carboxymethyl starch, carboxymethyl cellulose, and hydroxypropyl cellulose; nonionic types such as polyvinyl alcohol, and polyvinyl ethers; cationic types such as polyvinyl pyridine, polyvinyl morpholinone, and polyN,N-dimethylaminoethyl or polyN,N-diethylaminopropyl acrylates and methacrylates, and the respective quaternary salts thereof. Typically, hydrogel-forming absorbent polymers useful in the present invention have a multiplicity of anionic, functional groups, such as sulfonic acid, and more typically carboxy, groups. Examples of polymers suitable for use herein include those which are prepared from polymerizable, unsaturated, acid-containing monomers. Thus, such monomers include the olefinically unsaturated acids and anhydrides that contain at least



one carbon to carbon olefinic double bond. More specifically, these monomers can be selected from olefinically unsaturated carboxylic acids and acid anhydrides, olefinically unsaturated sulfonic acids, and mixtures thereof. The disclosure that follows describes preferred properties of the absorbent polymers useful herein. These properties should not be interpreted as limitations; rather, they merely indicate the progression that has occurred in the absorbent polymer art over the past several years.

Some non-acid monomers can also be included, usually in minor amounts, in preparing the hydrogel-forming absorbent polymers herein. Such non-acid monomers can include, for example, the water-soluble or water-dispersible esters of the acid-containing monomers, as well as monomers that contain no carboxylic or sulfonic acid groups at all. Optional non-acid monomers can thus include monomers containing the following types of functional groups: carboxylic acid or sulfonic acid esters, hydroxyl groups, amide-groups, amino groups, nitrile groups, quaternary ammonium salt groups, aryl groups (e.g., phenyl groups, such as those derived from styrene monomer). These non-acid monomers are well-known materials and are described in greater detail, for example, in U.S. Patent 4,076,663 (Masuda et al.), issued February 28, 1978, and in U.S. Patent 4,062,817 (Westerman), issued December 13, 1977, both of which are incorporated by reference.

Olefinically unsaturated carboxylic acid and carboxylic acid anhydride monomers include the acrylic acids typified by acrylic acid itself, methacrylic acid, ethacrylic acid, l-chloroacrylic acid, a-cyanoacrylic acid,  $\alpha$ -methylacrylic acid (crotonic acid), l-phenylacrylic acid,  $\alpha$ -acryloxypropionic acid, sorbic acid, l-chlorosorbic acid, angelic acid, cinnamic acid, p-chlorocinnamic acid,  $\alpha$ -styrylacrylic acid, itaconic acid, citraconic acid, mesaconic acid, glutaconic acid, aconitic acid, maleic acid, fumaric acid, tricarboxyethylene and maleic acid anhydride.

Olefinically unsaturated sulfonic acid monomers include aliphatic or aromatic vinyl sulfonic acids such as vinylsulfonic acid, allyl sulfonic acid, vinyl toluene sulfonic acid and styrene sulfonic acid; acrylic and methacrylic sulfonic acid such as sulfoethyl acrylate, sulfoethyl methacrylate, sulfopropyl acrylate, sulfopropyl methacrylate, 2-hydroxy-3-methacryloxypropyl sulfonic acid and 2-acrylamide-2-methylpropane sulfonic acid.

Preferred hydrogel-forming absorbent polymers for use in the present invention contain carboxy groups. These polymers include hydrolyzed starch-acrylonitrile graft copolymers, partially neutralized hydrolyzed starch-acrylonitrile graft copolymers, starch-

acrylic acid graft copolymers, partially neutralized starch-acrylic acid graft copolymers, saponified vinyl acetate-acrylic ester copolymers, hydrolyzed acrylonitrile or acrylamide copolymers, slightly crosslinked network of any of the foregoing copolymers, partially neutralized polyacrylic acid, and slightly crosslinked network polymers of partially neutralized polyacrylic acid. These polymers can be used either solely or in the form of a mixture of two or more different polymers. Examples of these polymer materials are disclosed in U.S. Patent 3,661,875, U.S. Patent 4,076,663, U.S. Patent 4,093,776, U.S. Patent 4,666,983, and U.S. Patent 4,734,478.

Most preferred polymer materials for use in making the hydrogel-forming absorbent polymers are slightly crosslinked network polymers of partially neutralized polyacrylic acids and starch derivatives thereof. (The terms slightly crosslinked network polymer, network crosslinked polymer and homogeneously crosslinked polymer are used interchangeably herein.) The slightly crosslinked network polymers are preferably crosslinked to an extent of about 0.01 mole% to about 2 mole%, preferably from 0.05 mole % to about 0.75 mole% of total moles of monomer. Suitable crosslinked network polymers are not limited to those polymers which are crosslinked to such a degree, as other polymers, with a lesser or higher degree of crosslinking may also function satisfactorily in an agglomerate or absorbent storage member of the present invention. Most preferably, the hydrogel-forming absorbent polymers comprise from about 50 to about 95%, preferably about 75%, neutralized, slightly crosslinked network, polyacrylic acid (i.e., poly (sodium acrylate/acrylic acid)). Network crosslinking renders the polymer substantially water-insoluble and, in part, determines the absorptive capacity and extractable polymer content characteristics of the hydrogel-forming absorbent polymers. Processes for network crosslinking these polymers and typical network crosslinking agents are described in greater detail in U.S. Patent 4,076,663.

While the hydrogel-forming absorbent polymer is preferably of one type (i.e., homogeneous), mixtures of polymers can also be used in the present invention. For example, mixtures of starch-acrylic acid graft copolymers and slightly crosslinked network polymers of partially neutralized polyacrylic acid can be used in the present invention.

The hydrogel-forming absorbent polymer component may also be in the form of a mixed-bed ion-exchange composition comprising a cation-exchange hydrogel-forming absorbent polymer and an anion-exchange hydrogel-forming absorbent polymer. Such mixed-bed ion-exchange compositions are described in, e.g., U.S. Patent Application

Serial No. 09/003,565, filed January 7, 1998 by Hird, et al. (titled "ABSORBENT POLYMER COMPOSITIONS HAVING HIGH SORPTION CAPACITIES UNDER AN APPLIED PRESSURE"); U.S. Patent Application Serial Nos. 09/003,905, filed January 7, 1998 and 09/130,321, filed August 7, 1998 by Ashraf, et al. (titled "ABSORBENT POLYMER COMPOSITIONS WITH HIGH  
5 SORPTION CAPACITY AND HIGH FLUID PERMEABILITY UNDER AN APPLIED PRESSURE"); U.S. Patent Application Serial No. 09/003,918, filed January 7, 1998 by Ashraf, et al. (titled "ABSORBENT POLYMER COMPOSITIONS HAVING HIGH SORPTION CAPACITIES UNDER AN APPLIED PRESSURE AND IMPROVED INTEGRITY IN THE SWOLLEN STATE"); and U.S. Patent Application Serial No. 09/258,890, filed March 1, 1999 by Hird, et al. (titled "ABSORBENT  
10 POLYMER COMPOSITIONS HAVING HIGH SORPTION CAPACITIES UNDER AN APPLIED PRESSURE"), the disclosure of each of which is incorporated herein by reference.

The hydrogel-forming absorbent polymers useful in the present invention can have a size, shape and/or morphology varying over a wide range. These polymers can be in the form of particles that do not have a large ratio of greatest dimension to smallest  
15 dimension (e.g., granules, pulverulents, interparticle aggregates, interparticle crosslinked aggregates, and the like) and can be in the form of fibers, sheets, films, foams, flakes and the like. The hydrogel-forming absorbent polymers can also comprise mixtures with low levels of one or more additives, such as for example powdered silica, surfactants, glue, binders, and the like. The components in this mixture can be physically and/or  
20 chemically associated in a form such that the hydrogel-forming polymer component and the non-hydrogel-forming polymer additive are not readily physically separable.

The hydrogel-forming absorbent polymers can be essentially non-porous (i.e., no internal porosity) or have substantial internal porosity.

For particles as described above, particle size is defined as the dimension  
25 determined by sieve size analysis. Thus, for example, a particle that is retained on a U.S.A. Standard Testing Sieve with 710 micron openings (e.g., No. 25 U.S. Series Alternate Sieve Designation) is considered to have a size greater than 710 microns; a particle that passes through a sieve with 710 micron openings and is retained on a sieve with 500 micron openings (e.g., No. 35 U.S. Series Alternate Sieve Designation) is  
30 considered to have a particle size between 500 and 710  $\mu\text{m}$ ; and a particle that passes through a sieve with 500 micron openings is considered to have a size less than 500  $\mu\text{m}$ . The mass median particle size of a given sample of hydrogel-forming absorbent polymer particles is defined as the particle size that divides the sample in half on a mass basis, i.e., one-half of the sample by weight will have a particle size less than the mass median

size and one-half of the sample will have a particle size greater than the mass median size. A standard particle-size plotting method (wherein the cumulative weight percent of the particle sample retained on or passed through a given sieve size opening is plotted versus sieve size opening on probability paper) is typically used to determine mass median particle size when the 50% mass value does not correspond to the size opening of a U.S.A. Standard Testing Sieve. These methods for determining particle sizes of the hydrogel-forming absorbent polymer particles are further described in U.S. Patent 5,061,259 (Goldman et al.), issued October 29, 1991, which is incorporated by reference.

For particles of hydrogel-forming absorbent polymers useful in the present invention, the particles will generally range in size from about 1 to about 2000  $\mu\text{m}$ , more preferably from about 20 to about 1000  $\mu\text{m}$ . The mass median particle size will generally be from about 20 to about 1500  $\mu\text{m}$ , more preferably from about 50  $\mu\text{m}$  to about 1000  $\mu\text{m}$ , and even more preferably from about 100 to about 800  $\mu\text{m}$ .

Where relatively high concentrations (e.g., 40-60% or greater, by weight) of hydrogel forming absorbent polymer are utilized in the absorbent members of the present invention, still other properties of the absorbent polymer may be relevant. In such embodiments, the materials may have one or more of the properties described by U.S. Patent No. 5,562,646, issued Oct. 8, 1996 to Goldman et al. and U.S. Patent No. 5,599,335, issued Feb. 4, 1997 to Goldman et al., the disclosure of each of which is incorporated by reference herein.

## 2. Methods for Making

The basic hydrogel-forming absorbent polymer can be formed in any conventional manner. Typical and preferred processes for producing these polymers are described in U.S. Reissue Patent 32,649 (Brandt et al.), issued April 19, 1988, U.S. Patent 4,666,983 (Tsubakimoto et al.), issued May 19, 1987, and U.S. Patent 4,625,001 (Tsubakimoto et al.), issued November 25, 1986, all of which are incorporated by reference. The hydrogel-forming absorbent polymer may also be purchased commercially in a ready to use form.

Preferred methods for forming the basic hydrogel-forming absorbent polymer are those involving aqueous solution or other solution polymerization methods. As described in the above-referenced U.S. Patent Reissue 32,649, aqueous solution polymerization involves the use of an aqueous reaction mixture to carry out polymerization. The aqueous reaction mixture is then subjected to polymerization conditions which are

sufficient to produce in the mixture, substantially water-insoluble, slightly crosslinked network polymer or homogeneously crosslinked polymer (the terms slightly crosslinked network polymer, crosslinked network polymer and homogeneously crosslinked polymer are used interchangeably herein). The mass of polymer formed can then be pulverized  
5 or chopped to form individual particles.

More specifically, the aqueous solution polymerization method for producing the hydrogel-forming absorbent polymer comprises the preparation of an aqueous reaction mixture in which to carry out the polymerization. One element of such a reaction mixture is the acid group-containing monomer that will form the "backbone" of the hydrogel-  
10 forming absorbent polymer to be produced. The reaction mixture will generally comprise about 100 parts by weight of the monomer. Another component of the aqueous reaction mixture comprises a network crosslinking agent. Network crosslinking agents useful in forming the hydrogel-forming absorbent polymer according to the present invention are described in more detail in the above-referenced U.S. Reissue Patent 32,649, U.S.  
15 Patent 4,666,983, and U.S. Patent 4,625,001. The network crosslinking agent will generally be present in the aqueous reaction mixture in an amount of from about 0.001 mole percent to about 5 mole percent based on the total moles of monomer present in the aqueous mixture (about 0.01 to about 20 parts by weight, based on 100 parts by  
20 weight of the monomer). An optional component of the aqueous reaction mixture comprises a free radical initiator including, for example, peroxygen compounds such as sodium, potassium, and ammonium persulfates, caprylyl peroxide, benzoyl peroxide, hydrogen peroxide, cumene hydroperoxides, tertiary butyl diperphthalate, tertiary butyl perbenzoate, sodium peracetate, sodium percarbonate, and the like. Other optional  
25 components of the aqueous reaction mixture comprise the various non-acidic co-monomers, including esters of the essential unsaturated acidic functional group-containing monomers or other co-monomers containing no carboxylic or sulfonic acid functionalities at all.

The aqueous reaction mixture is subjected to polymerization conditions which are sufficient to produce in the mixture substantially water-insoluble, but water-swella-  
30 ble, hydrogel-forming absorbent slightly crosslinked network polymers. The polymerization conditions are also discussed in more detail in the three above-referenced patents. Such polymerization conditions generally involve heating (thermal activation techniques) to a polymerization temperature from about 0° to about 100°C, more preferably from about 5° to about 40°C. Polymerization conditions under which the aqueous reaction

mixture is maintained can also include, for example, subjecting the reaction mixture, or portions thereof, to any conventional form of polymerization activating irradiation. Radioactive, electronic, ultraviolet, or electromagnetic radiation are alternative conventional polymerization techniques.

5       The acid functional groups of the hydrogel-forming absorbent polymer formed in the aqueous reaction mixture are also preferably neutralized. Neutralization can be carried out in any conventional manner that results in at least about 25 mole percent, and more preferably at least about 50 mole percent, of the total monomer utilized to form the polymer being acid group-containing monomers that are neutralized with a salt-  
10       forming cation. Such salt-forming cations include, for example, alkali metals, ammonium, substituted ammonium and amines as discussed in further detail in the above-references U.S. Reissue Patent 32,649.

      While it is preferred that the particulate versions of hydrogel-forming absorbent polymer be manufactured using an aqueous solution polymerization process, it is also  
15       possible to carry out the polymerization process using multi-phase polymerization processing techniques such as inverse emulsion polymerization or inverse suspension polymerization procedures. In the inverse emulsion polymerization or inverse suspension polymerization procedures, the aqueous reaction mixture as described before is suspended in the form of tiny droplets in a matrix of a water-immiscible, inert  
20       organic solvent such as cyclohexane. The resultant particles of hydrogel-forming absorbent polymer are generally spherical in shape. Inverse suspension polymerization procedures are described in greater detail in U.S. Patent 4,340,706 (Obaysashi et al.), issued July 20, 1982, U.S. Patent 4,506,052 (Flesher et al.), issued March 19, 1985, and U.S. Patent 4,735,987 (Morita et al.), issued April 5, 1988, all of which are incorporated  
25       by reference.

      Surface crosslinking of the initially formed polymers is a preferred process for obtaining hydrogel-forming absorbent polymers having relatively high porosity hydrogel-layer ("PHL"), performance under pressure ("PUP") capacity and saline flow conductivity ("SFC") values, which may be beneficial in the context of the present invention. Suitable  
30       general methods for carrying out surface crosslinking of hydrogel-forming absorbent polymers according to the present invention are disclosed in U.S. Patent 4,541,871 (Obayashi), issued September 17, 1985; published PCT application WO92/16565 (Stanley), published October 1, 1992, published PCT application WO90/08789 (Tai), published August 9, 1990; published PCT application WO93/05080 (Stanley); published

March 18, 1993; U.S. Patent 4,824,901 (Alexander), issued April 25, 1989; U.S. Patent 4,789,861 (Johnson), issued January 17, 1989; U.S. Patent 4,587,308 (Makita), issued May 6, 1986; U.S. Patent 4,734,478 (Tsubakimoto), issued March 29, 1988; U.S. Patent 5,164,459 (Kimura et al.), issued November 17, 1992; published German Patent  
5 Application 4,020,780 (Dahmen), published August 29, 1991; and published European Patent Application 509,708 (Gartner), published October 21, 1992; all of which are incorporated by reference. See also, U.S. Patent 5,562,646 (Goldman et al.), issued Oct. 8, 1996 and U.S. Patent 5,599,335 (Goldman et al.), issued Feb. 4, 1997.

The hydrogel-forming absorbent polymer particles prepared according to the  
10 present invention are typically substantially dry. The term "substantially dry" is used herein to mean that the particles have a liquid content, typically water or other solution content, less than about 50%, preferably less than about 20%, more preferably less than about 10%, by weight of the particles. In general, the liquid content of the hydrogel-forming absorbent polymer particles is in the range of from about 0.01% to about 5% by  
15 weight of the particles. The individual particles can be dried by any conventional method such as by heating. Alternatively, when the particles are formed using an aqueous reaction mixture, water can be removed from the reaction mixture by azeotropic distillation. The polymer-containing aqueous reaction mixture can also be treated with a dewatering solvent such as methanol. Combinations of these drying procedures can  
20 also be used. The dewatered mass of polymer can then be chopped or pulverized to form substantially dry particles of the hydrogel-forming absorbent polymer.

B. High Surface Area Open-Celled Hydrophilic Foam

In addition to the particulate hydrogel-forming absorbent polymer, the agglomerates of the high capillary suction storage absorbent members of the present  
25 invention comprise particulate high surface area open-celled hydrophilic foam. It is this high surface area material that provides, in combination with the hydrogel-forming absorbent polymer, the agglomerates and members with high capillary sorption absorbent capacity. As discussed herein, high surface area materials are described, at least in one regard, in terms of their capillary sorption absorbent capacity (measured  
30 without hydrogel-forming polymer or any other optional material contained in the actual storage absorbent member, such as adhesives, bonding agents, etc.). It is recognized that materials having high surface areas may have uptake capacities at very high suction heights (e.g., 100 cm or higher). This allows the high surface area materials to provide one or both of the following functions: i) a capillary pathway of liquid to the osmotic

absorbents, and/or ii) additional absorbent capacity. Thus, while the high surface area materials may be described in terms of their surface area per weight or volume, applicants herein alternatively use capillary sorption absorbent capacity to describe the high surface area material because capillary sorption absorbent capacity is a performance parameter that generally will provide the absorbent members of the present invention with the requisite suction capabilities to provide improved absorbent articles. It will be recognized that certain high surface area materials, e.g., glass microfibers, will themselves not exhibit particularly high capillary sorption absorbent capacity at all heights, especially very high heights (e.g., 100 cm and higher). Nonetheless, such materials may provide the desired capillary pathway of liquid to the hydrogel-forming absorbent polymer or other osmotic absorbent to provide the requisite capillary sorption absorbent capacities, even at relatively high heights, when combined with the hydrogel-forming polymer or other osmotic absorbent.

The term "high surface area material" refers to any material that itself (i.e., as measured without any other optional material that makes up the storage absorbent member) exhibits one or more of the following capillary sorption absorbent capacities: (I) A capillary sorption absorbent capacity of at least about 2 g/g at a suction height of 100 cm, preferably at least about 3 g/g, still more preferably at least about 4 g/g, and still more preferably at least about 6 g/g, at a height of 100 cm; (II) A capillary sorption absorbent capacity at a height of 35 cm of at least about 5 g/g, preferably at least about 8 g/g, more preferably at least about 12 g/g; (III) A capillary sorption absorbent capacity at a height of 50 cm of at least about 4 g/g, preferably at least about 7 g/g, more preferably at least about 9 g/g; (IV) A capillary sorption absorbent capacity at a height of 140 cm of at least about 1 g/g, preferably at least about 2 g/g, more preferably at least about 3 g/g, still more preferably at least about 5 g/g; or (V) A capillary sorption absorbent capacity at a height of 200 cm of at least about 1 g/g, preferably at least about 2 g/g, more preferably at least about 3 g/g, still more preferably at least about 5 g/g.

The high surface area open-celled hydrophilic foams useful herein are described in some respects below in terms of their physical properties. To measure certain of these properties, it is necessary to perform analysis on the foam in sheet form. Thus, insofar as a foam is used in particulate form and is prepared from a previously formed sheet, physical property measurements will be conducted on the sheet foam (i.e., prior to forming particulates). Where the foam is formed in situ into particles (or beads) during the polymerization process, a similar foam (in terms of chemical composition, cell-size,



W:O ratio, etc.) can be formed into sheets for the purpose of making such measurements.

1. General Foam Characteristics

High surface area open-celled hydrophilic foams useful in agglomerates of the  
5 high capillary suction storage absorbent members of the present invention are known in  
the art. Particularly preferred foams are those obtained by polymerizing a high internal  
phase water-in-oil emulsion ("HIPE"), such as those described in U.S. Patent  
No. 5,387,207 and U.S. Patent No. 5,650,222. A particularly preferred high surface area  
open-celled hydrophilic foam is one that is thin-until-wet, as described in detail U.S.  
10 Patent No. 5,387,207, hereby incorporated by reference. Other particularly preferred  
high surface area open-celled hydrophilic foams are described in more detail in co-  
pending U.S. Patent Application Serial No. 09/042,429, filed March 13, 1998 by T. A.  
DesMarais) (titled "HIGH SUCTION POLYMERIC FOAM MATERIALS") and U.S. Patent  
No. 6,013,589 (T. A. DesMarais et al.), issued January 1, 2000, the disclosure of each  
15 of which is incorporated by reference herein. (Specific preferred foams described in one  
or both of these copending applications are described in the Examples section below.)

Another material that may be incorporated into the HIPE foam structure is a  
20 hydratable, and preferably hygroscopic or deliquescent, water soluble inorganic salt.  
Such salts include, for example, toxicologically acceptable alkaline earth metal salts.  
Materials of this type and their use in conjunction with oil-soluble surfactants as the foam  
hydrophilizing agent is described in greater detail in Application Serial No. 07/743,951,  
filed Aug. 12, 1991. Preferred salts of this type include the calcium halides such as  
calcium chloride which, as previously noted, can also be employed as the electrolyte in  
the water phase of the HIPE emulsions used to prepare the high surface area open-  
25 celled hydrophilic foams. The hydratable salt will be present in the residual water of the  
high surface area open-celled hydrophilic foam. In addition to keeping the residual water  
in the foam structure from evaporating, these hydratable salts can also increase the  
surface tension of the residual water. The hydratable salts will be present in the foam  
structure of the present invention in an amount of at least about 0.1% by weight of the  
30 foam, and typically in the range of from about 0.1% to about 8%, preferably from about  
2% to about 6%, most preferably from about 4% to about 6%, by weight of the foam.

High surface area hydrophilic foams useful herein are those which are relatively  
open-celled. This means many of the individual cells of the foam are in unobstructed  
communication with adjoining cells. The cells in such relatively open-celled foam

structures have intercellular openings or "windows" that are large enough to permit ready liquid transfer from one cell to the other within the foam structure.

These relatively open-celled foam structures will generally have a reticulated character with the individual cells being defined by a plurality of mutually connected, three dimensionally branched webs. The strands of foam material making up these branched webs can be referred to as "struts." For purposes of the present invention, a most preferred foam material will have at least about 80% of the cells in the foam structure that are at least 1  $\mu\text{m}$  in size in liquid communication with at least one adjacent cell.

In addition to being open-celled, these high surface area foams are sufficiently hydrophilic to permit the foam to absorb aqueous liquids. The internal surfaces of the foam structures are rendered hydrophilic by residual hydrophilizing surfactants left in the foam structure after polymerization, or by selected post-polymerization foam treatment procedures, as described hereafter.

The extent to which these high surface area open-celled foams are "hydrophilic" can be quantified by the "adhesion tension" value exhibited when in contact with an absorbable test liquid. The adhesion tension exhibited by these foams can be determined experimentally using a procedure where weight uptake of a test liquid, e.g., synthetic urine, is measured for a sample of known dimensions and capillary suction specific surface area. Such a procedure is described in greater detail in the Test Methods section of U.S. Patent 5,387,207, *infra*. Foams which are useful high surface area materials in the present invention are generally those which exhibit an adhesion tension value of from about 15 to about 65 dynes/cm, more preferably from about 20 to about 65 dynes/cm, as determined by capillary absorption of synthetic urine having a surface tension of  $65 \pm 5$  dynes/cm.

The high surface area open-celled hydrophilic foams useful herein are preferably prepared in the form of collapsed (i.e., unexpanded), high surface area open-celled hydrophilic foams that, upon contact with aqueous liquids, absorb such liquids and expand when the amount absorbed lowers the combined capillary pressure plus confining pressure to below the expansion pressure (described below) of the foam. These collapsed high surface area open-celled hydrophilic foams are usually obtained by expressing the water phase from the polymerized HIPE foam through compressive forces, and/or thermal drying and/or vacuum dewatering. After compression, and/or

thermal drying/vacuum dewatering, these high surface area open-celled hydrophilic foams are in a collapsed, or unexpanded state.

The cellular structure of a representative collapsed HIPE foam from which water has been expressed by compression is shown in the photomicrograph of Figs. 3 and 4 of U.S. Patent No. 5,650,222, discussed above. As shown in these figures, the cellular structure of the foam is distorted, especially when compared to the expanded HIPE foam structures shown in Figs. 1 and 2 of the '222 patent. As can also be seen in Figs. 3 and 4 of the '222 patent, the voids or pores (dark areas) in the collapsed foam structure have been flattened or elongated. (It is noted that the foams depicted in the '222 patent are in sheet form. The preparation of this particular foam and related foams are described herein in Examples 1 through 3, and these very high surface area foams are described in more detail in co-pending U.S. Patent Application Serial No. 09/042,429, filed March 13, 1998 by T. A. DesMarais, and U.S. Patent No. 6,013,589 (T. A. DesMarais), issued January 11, 2000, the disclosure of each of which is incorporated by reference herein.

Following compression and/or thermal drying/vacuum dewatering, the collapsed high surface area open-celled hydrophilic foam may re-expand when wetted with aqueous liquids. Surprisingly, these high surface area open-celled hydrophilic foams remain in this collapsed, or unexpanded, state for significant periods of time, e.g., up to at least about 1 year. The ability of these high surface area open-celled hydrophilic foams to remain in this collapsed/unexpanded state is believed to be due to capillary forces, and in particular the capillary pressures developed within the foam structure. As used herein, "capillary pressures" refers to the pressure differential across the liquid/air interface due to the curvature of meniscus within the narrow confines of the pores in the foam. [See Chatterjee, "Absorbency," Textile Science and Technology, Vol. 7, 1985, p. 36.]

After compression, and/or thermal drying/vacuum dewatering to a practicable extent, these high surface area open-celled hydrophilic foams have residual water that includes both the water of hydration associated with the hygroscopic, hydrated salt incorporated therein, as well as free water absorbed within the foam. This residual water (assisted by the hydrated salts) is believed to exert capillary pressures on the resulting collapsed foam structure. Collapsed high surface area open-celled hydrophilic foams of the present invention can have residual water contents of at least about 4%, typically from about 4 to about 40%, by weight of the foam when stored at ambient conditions of

72°F (22°C) and 50% relative humidity. Preferred collapsed high surface area open-celled hydrophilic foams have residual water contents of from about 5 to about 30% by weight of the foam.

5 A key parameter of these foams is their glass transition temperature. The T<sub>g</sub> represents the midpoint of the transition between the glassy and rubbery states of the polymer. Foams that have a higher T<sub>g</sub> than the temperature of use can be very strong but will also be rigid and potentially prone to fracture. Such foams also typically take a long time to recover to the expanded state when wetted with aqueous liquids colder than the T<sub>g</sub> of the polymer after having been stored in the collapsed state for prolonged  
10 periods. The desired combination of mechanical properties, specifically strength and resilience, typically necessitates a fairly selective range of monomer types and levels to achieve these desired properties.

For foams useful in the present invention, the T<sub>g</sub> should be as low as possible, so long as the foam has acceptable strength at in-use temperatures. Accordingly,  
15 monomers are selected as much as possible that provide corresponding homopolymers having lower T<sub>g</sub>'s. It has been found that the chain length of the alkyl group on the acrylate and methacrylate comonomers can be longer than would be predicted from the T<sub>g</sub> of the homologous homopolymer series. Specifically, it has been found that the homologous series of alkyl acrylate or methacrylate homopolymers have a minimum T<sub>g</sub>  
20 at a chain length of 8 carbon atoms. By contrast, the minimum T<sub>g</sub> of the copolymers of the present invention occurs at a chain length of about 12 carbon atoms. (While the alkyl substituted styrene monomers can be used in place of the alkyl acrylates and methacrylates, their availability is currently extremely limited).

The shape of the glass transition region of the polymer can also be important,  
25 i.e., whether it is narrow or broad as a function of temperature. This glass transition region shape is particularly relevant where the in-use temperature (usually ambient or body temperature) of the polymer is at or near the T<sub>g</sub>. For example, a broader transition region can mean an incomplete transition at in-use temperatures. Typically, if the transition is incomplete at the in-use temperature, the polymer will evidence greater  
30 rigidity and will be less resilient. Conversely, if the transition is completed at the in-use temperature, then the polymer will exhibit faster recovery from compression when wetted with aqueous liquids. Accordingly, it is desirable to control the T<sub>g</sub> and the breadth of the transition region of the polymer to achieve the desired mechanical properties. Generally, it is preferred that the T<sub>g</sub> of the polymer be at least about 10°C lower than the in-use

temperature. (The  $T_g$  and the width of the transition region are derived from the loss tangent vs. temperature curve from a dynamic mechanical analysis (DMA) measurement, as described in the Test Methods section of U.S. Patent No. 5,650,222).

## 2. Capillary Suction Specific Surface Area

5 While the high surface area materials in general have been described in terms of their capillary sorption absorbent capacity, the high surface area open-celled hydrophilic foams useful herein may also be described in terms of their capillary suction specific surface area (hereafter referred to as "CSSSA"). In general, CSSSA is a measure of the test-liquid-accessible surface area of the open-celled hydrophilic network forming a particular foam per unit mass of the bulk foam material (polymer structural material plus solid residual material). Capillary suction specific surface area is determined both by the dimensions of the cellular units in the foam and by the density of the polymer, and is thus a way of quantifying the total amount of solid surface provided by the foam network to the extent that such a surface participates in absorbency. For purposes of characterizing the foams useful herein, CSSSA is measured on a sheet of the foam in question.

10 The CSSSA of a foam is particularly relevant to whether the foam will provide the requisite capillary suction for use in preparing agglomerates and storage absorbent members of the present invention. This is because the capillary pressure developed within the foam structure is proportional to the capillary suction specific surface area. In addition, the CSSSA is relevant to whether adequate capillary pressures are developed within the foam structure to keep it in a collapsed state until wetted with aqueous liquids. Assuming other factors such as the foam density and adhesion tension are constant, this means that, as the CSSSA is increased (or decreased), the capillary pressure within the foam structure also increases (or decreases) proportionately.

25 For purposes of the present invention, CSSSA is determined by measuring the amount of capillary uptake of a low surface tension liquid (e.g., ethanol) which occurs within a foam sample of a known mass and dimensions. A detailed description of such a procedure for determining foam specific surface area is set forth in the Test Methods section of U.S. Patent 5,387,207, which is incorporated by reference. Any reasonable alternative method for determining CSSSA can also be utilized.

30 The collapsed high surface area open-celled hydrophilic foams of the present invention useful as absorbents are those that have a CSSSA of at least about 3 m<sup>2</sup>/g.

Typically, the CSSSA is in the range from about 3 to about 30 m<sup>2</sup>/g, preferably from about 4 to about 17 m<sup>2</sup>/g, most preferably from about 5 to about 15 m<sup>2</sup>/g. Foams having

such CSSSA values (with expanded state densities of from about 0.010 to about 0.033 g/cc) will generally possess an especially desirable balance of absorbent capacity, liquid-retaining and liquid-wicking or distribution characteristics for aqueous liquids such as urine. In addition, foams having such CSSSA can develop a sufficient capillary pressure to keep the foam in a collapsed, unexpanded state until wetted with such aqueous liquids.

### 3. Capillary Pressures and Forces Within Foam Structure

As discussed above, for particularly preferred collapsible high surface area open-celled hydrophilic foams, in their collapsed state the capillary pressures developed within the foam structure at least equal the forces exerted by the elastic recovery or modulus of the compressed polymer. In other words, the capillary pressure necessary to keep the collapsed foam relatively thin is determined by the countervailing force exerted by the compressed high surface area open-celled hydrophilic foam as it tries to "spring back." The elastic recovery tendency of high surface area open-celled hydrophilic foams can be estimated from stress-strain experiments where the expanded foam is compressed to about 1/6 (17%) of its original, expanded thickness and then held in this compressed state until a relaxed stress value is measured. Alternatively, and for the purposes of the present invention, the relaxed stress value is estimated from measurements on the high surface area open-celled hydrophilic foam in its collapsed state when in contact with aqueous liquids, e.g., water. This alternative relaxed stress value is hereafter referred to as the "expansion pressure" of the foam. The expansion pressure for collapsed high surface area open-celled hydrophilic foams of the present invention is about 50 kiloPascals (kPa) or less and typically from about 7 to about 40 kPa. A detailed description of a procedure for estimating the expansion pressure of foams is set forth in the Test Methods section of U.S. Patent 5,387,207.

### 4. Free Absorbent Capacity

Another important property of the high surface area open-celled hydrophilic foams useful in the present invention is their free absorbent capacity. "Free absorbent capacity" (or "FAC") is the total amount of test liquid (synthetic urine) which a given foam sample will absorb into its cellular structure per unit mass of solid material in the sample. To be especially useful in the agglomerates and storage absorbent members of the present invention, the high surface area open-celled hydrophilic foams should have a free absorbent capacity of from about 30 to about 100 mL, preferably from about 30 to about 75 mL of synthetic urine per gram of dry foam material. The procedure for

determining the free absorbent capacity of the foam is described hereafter in the Test Methods section of U.S. Patent No. 5,650,222.

5. Expansion Factor

Upon exposure to aqueous liquids, preferred collapsed high surface area open-celled hydrophilic foams absorb the liquids and expand. The high surface area open-celled hydrophilic foams, in their expanded state, absorb more liquid than most other foams. The "expansion factor" for these foams is at least about 4X, i.e. the thickness of the foam in its expanded state is at least about 4 times the thickness of the foam in its collapsed state. The collapsed foams preferably have an expansion factor in the range of from about 4X to about 15X, more preferably from about 5X to about 10X.

For the purposes of the present invention, the relationship between expanded and collapsed thickness for compressively dewatered foams can be empirically predicted from the following equation:

$$\text{thickness expanded} = \text{thickness collapsed} \times ((0.133 \times \text{W:O ratio}) \pm 2)$$
  
where: thickness expanded is the thickness of the foam in its expanded state; thickness collapsed is the thickness of the foam in its collapsed state; and W:O ratio is the water-to-oil ratio of the HIPE from which the foam is made.

Thus, a typical high surface area open-celled hydrophilic foam made from an emulsion with a water-to-oil ratio of 60:1 would have a predicted expansion factor of 8.0, i.e., an expanded thickness 8 times the collapsed thickness of the foam. The procedure for measuring the expansion factor is described hereafter in the Test Methods section of U.S. Patent 5,650,222.

6. Resistance to Compression Deflection

A relevant mechanical feature of the high surface area open-celled hydrophilic foams useful in the present invention is their strength in their expanded state, as determined by resistance to compression deflection (RTCD). The RTCD exhibited by the foams herein is a function of the polymer modulus, as well as the density and structure of the foam network. The polymer modulus is, in turn, determined by: a) the polymer composition; b) the conditions under which the foam is polymerized (for example, the completeness of polymerization obtained, specifically with respect to crosslinking); and c) the extent to which the polymer is plasticized by residual material, e.g., emulsifiers, left in the foam structure after processing.

To be useful as the high surface area portion of the absorbent members of the present invention, the high surface area open-celled hydrophilic foams should be suitably

resistant to deformation or compression by forces encountered in use. Foams which do not possess sufficient foam strength in terms of RTCD may provide the requisite capillary suction capacity under no-load conditions but will not provide those capacities under the compressive stress caused by the motion and activity of the user of the absorbent articles that contain the foam.

The RTCD exhibited by the high surface area open-celled hydrophilic foams useful in the present invention can be quantified by determining the amount of strain produced in a sample of saturated foam held under a certain confining pressure for a specified temperature and period of time. The method for carrying out this particular type of test is described hereafter in the Test Methods section of U.S. Patent No. 5,650,222. Foams useful herein will preferably exhibit a RTCD such that a confining pressure of 5.1 kPa produces a strain of typically about 90% or less compression of the foam structure when it has been saturated to its free absorbent capacity with synthetic urine having a surface tension of  $65 \pm 5$  dynes/cm. Preferably the strain produced under such conditions will be in the range from about 1 to about 90%, more preferably from about 1 to about 25%, still more preferably from about 2 to about 10%, still more preferably from about 2 to about 5%.

#### 7. Vertical Hang Sorption Height

The high surface area open-celled hydrophilic foams useful herein can be also be described in terms of their vertical hang sorption height (hereafter "VHSH"). The VHSH height at X % is the height in cm where X % of the 0 cm capacity (or FAC) is retained in the foam. A typical value of importance is the VHSH at 90%, though in principle X may be any value. The most reproducible measure for VHSH is achieved at X = 90%, within the experience of the inventors. It will be obvious to one skilled in the art that this single point value does not fully express the shape of the curve obtained in a plot of capacity vs. height. The single point however serves as a practical point of comparison for the foams useful herein. In this regard, the foams will typically have an equilibrium 90% VHSH of at least about 20 cm, preferably at least about 40 cm, still more preferably at least about 60 cm, still more preferably at least about 70 cm and still more preferably at least about 80 cm. Typically, preferred high surface area open-celled hydrophilic foams will have a 90% VHSH of from about 20 to about 90 cm, more typically from about 60 to about 90 cm, more typically from about 70 to about 90 cm, still more typically from, about 80 to about 90 cm. The method for measuring 90% VHSH is described in detail in the Test Methods section below. As indicated, where the high surface area open-celled



hydrophilic foam is in particulate form when combined into an agglomerate of the present invention, 90% VSH is measured on the corresponding foam in sheet form (i.e., prior to forming particulates). Where the foam is formed into particles (or beads) during the polymerization process, a similar foam can be formed into sheets for assessing the foam's 90% VSH.

8. Other Properties of High Surface Area Open-Celled Hydrophilic Foam

Foam cells, and especially cells that are formed by polymerizing a monomer-containing oil phase that surrounds relatively monomer-free water-phase droplets, will frequently be substantially spherical in shape. The size or "diameter" of such spherical cells is a commonly used parameter for characterizing foams in general. Since cells in a given sample of high surface area open-celled hydrophilic foam will not necessarily be of approximately the same size, an average cell size, i.e., average cell diameter, will often be specified.

A number of techniques are available for determining the average cell size of foams. The most useful technique, however, for determining cell size in foams involves a simple measurement based on the scanning electron photomicrograph of a foam sample.

The cell size measurements given herein are based on the number average cell size of the foam in its expanded state, e.g., as shown in Fig. 1 of U.S. Patent No. 5,650,222. The foams useful in accordance with the present invention will preferably have a number average cell size of about 80  $\mu\text{m}$  or less, and typically from about 5 to about 50  $\mu\text{m}$ .

"Foam density" (i.e., in grams of foam per cubic centimeter of foam volume in air) is specified herein on a dry basis. The amount of absorbed water-soluble residual materials, e.g., residual salts and liquid left in the foam, for example, after HIPE polymerization, washing and/or hydrophilization, is disregarded in calculating and expressing foam density. Foam density does include, however, other water-insoluble residual materials such as emulsifiers present in the polymerized foam. Such residual materials can, in fact, contribute significant mass to the foam material.

Any suitable gravimetric procedure that will provide a determination of mass of solid foam material per unit volume of foam structure can be used to measure foam density. For example, an ASTM gravimetric procedure described more fully in the Test Methods section of U.S. Patent No. 5,387,207 (Dyer et al.) issued Feb. 7, 1995, *supra*, is one method that can be employed for density determination. In their collapsed state,

high surface area open-celled hydrophilic foams useful in the present invention have dry basis density values (exclusive of any residual salts and/or water) in the range of from about 0.1 to about 0.2 g/cc, preferably from about 0.11 to about 0.19 g/cc, and most preferably from about 0.12 to about 0.17 g/cc. In their expanded state, high surface area  
5 open-celled foams useful herein will have dry basis density values in the range of from about 0.01 to about 0.033 g/cc, preferably from about 0.013 to about 0.033 g/cc.

Vertical wicking, i.e., liquid wicking in a direction opposite from gravitational force, is a desirable performance attribute for high surface area open-celled hydrophilic foams useful herein. For the purposes of this invention, vertical wicking rate is reflective of the  
10 permeability of the material, and thus, the ability of the material to deliver liquid to the hydrogel-forming absorbent polymer.

Vertical wicking rate is determined by measuring the time taken for a colored test liquid (e.g., synthetic urine) in a reservoir to wick a vertical distance of 5 cm through a test strip of foam of specified size. The vertical wicking procedure is described in greater  
15 detail in the Test Methods section of U.S. Patent No. 5,387,207, but is performed at 31°C, instead of 37°C. To be especially useful in absorbent members for absorbing urine, the foams useful herein will preferably wick synthetic urine (65 + 5 dynes/cm) to a height of 5 cm in no more than about 15 minutes. More preferably, the preferred foam absorbents of the present invention wick synthetic urine to a height of 5 cm in no more  
20 than about 10 minutes.

The vertical wicking absorbent capacity test measures the amount of test liquid per gram of absorbent foam that is held within each one in. (2.54 cm) vertical section of the same standard size foam sample used in the vertical wicking test. Such a determination is generally made after the sample has been allowed to vertically wick test  
25 liquid to equilibrium (e.g., after about 18 hours). Like the vertical wicking test, the vertical wicking absorbent capacity test is described in greater detail in the Test Methods section of U.S. Patent No. 5,387,207 (Dyer et al.) issued Feb. 7, 1995, *supra*. High vertical wicking absorbent capacities at high heights are theoretically equivalent to high capillary sorption absorbent capacities at high heights. Since the sheet form of the foams useful  
30 herein is amenable to the former test and the former test is more easily and cheaply performed, the data from the former test are recommended as the means of characterizing this important parameter of the foams of this invention.

While the high surface area open-celled hydrophilic foam is in a particulate form when incorporated into the agglomerate of the absorbent member, the foam may initially

be prepared in sheet form, and these sheets may be processed to provide particles of foam which are then combined with the hydrogelling polymer. As discussed above, the foams useful herein, and processes for their preparation, are described in great detail in U.S. Patent No. 5,387,207, U.S. Patent No. 5,650,222, co-pending U.S. Patent Application Serial No. 09/042,429 by T. A. DesMarais (titled "HIGH SUCTION POLYMERIC FOAM MATERIALS"), and U.S. Patent No. 6,013,589 (T. A. DesMarais et al.), issued January 11, 2000. Foam particles may be prepared by first forming a sheet of foam per the teachings of these references, followed by mechanical processing the foam to provide particles (e.g., pulverizing, cutting, chopping, etc.) of the desired dimension. Alternatively, foam particles may be prepared directly from emulsion in the form of polymeric microbeads, as described in U.S. Patent 5,653,922, issued Aug. 5, 1997 to Li et al., and U.S. Patent 5,583,162, issued Dec. 10, 1996 to Li et al., the disclosure of each of which is incorporated by reference herein. Specific embodiments for making agglomerates of the invention comprising particulate hydrogel-forming polymer and particulate high surface area open-celled hydrophilic foam are discussed in more detail below.

When the high surface area open-celled hydrophilic foam is in particulate form, the particle size of discrete foam particles can be very large or very small, or a mixture of various particle sizes, and depends in part on the size of the particulate hydrogel-forming absorbent polymer. Dry particle sizes can be greater than about 1000 microns, however it is believed that particles larger than about 1000 microns do not maintain optimum fluid delivery contact with the osmotic absorbent. It is believed that particles, measured when dry, which are less than about 1000 microns in size provide excellent fluid delivery contact with the osmotic absorbent. Dry particles sizes can also be greater than about 50 microns. It is believed that for a mass median particle size of dry particles smaller than about 50 microns, insufficient cellular structure of the high surface area open-celled hydrophilic foam exists to provide the delivery of fluid at high suction to the particulate hydrogel-forming absorbent polymer. In typical applications, dry particle size can be less than about 600 microns mass median particle size of dry particles, and typically between about 50 and 600 micron mass median particle size of dry particles. The dry particle size and mass median particle size of dry particles can be established by dry sieving the dry high surface area open-celled hydrophilic foam through the appropriate screens as is discussed previously herein in section III.A.1. in regard to dry sieving hydrogel-forming absorbent polymer particles.

A preferred method for dewatering particles of the high surface area open-celled hydrophilic foam is the unidirectional dewatering method, wherein water contained within wet particles while the particles are being made is removed in a single direction from each particle. The vacuum dewatering method of Examples 1-3, set forth below, is one example of unidirectional dewatering. It is believed that unidirectional dewatering of preferred particulate high surface area open-celled hydrophilic foam materials results in greater expansion of the materials when wetted than would otherwise be provided without unidirectional dewatering, and that the expansion provides additional contact of the high surface area open-celled hydrophilic foam and hydrogel-forming absorbent polymer as the hydrogel-forming absorbent polymer absorbs fluid from the high surface area open-celled hydrophilic foam and as the foam absorbs fluid.

C. Agglomerates Comprising Hydrogel-Forming Absorbent Polymer and High Surface Area Open-Celled Hydrophilic Foam

The agglomerates of the present invention comprise hydrogel-forming absorbent polymer particles and particles of high surface area open-celled hydrophilic foam. The agglomerates will preferably comprise at least about 1%, by weight (on an aggregate basis), high surface area open-celled hydrophilic foam. The agglomerate will preferably comprise at least about 10%, by weight, more preferably at least about 15%, by weight, still more preferably at least about 20%, by weight, high surface area open-celled hydrophilic foam. Typically, such agglomerates will comprise from about 1 to about 98%, by weight, more typically from about 10 to about 90%, by weight, still more typically from about 15 to about 85%, by weight, still more typically from about 20 to about 80%, and still more typically from about 20 to about 50%, by weight, of the high surface area open-celled hydrophilic foam material. Of course, the relative levels of the hydrogel-forming absorbent polymer and high surface area open-celled hydrophilic foam in the agglomerate will be dictated by, for example, the absorptive capacity of the hydrogel-forming absorbent polymer, the specific high surface area open-celled hydrophilic foam used, the nature of the high surface area open-celled hydrophilic foam (e.g., particle size), etc. To achieve the requisite level of capillary suction discussed above, there must be sufficient high surface area open-celled hydrophilic foam to provide suction capacity. Without wishing to be bound by theory, it is believed that three primary properties of the preferred collapsible high surface area open-celled hydrophilic foam materials described above allow these foams to function particularly effectively in high suction storage absorbent members. These three properties are: (i) relatively low density, (ii) the ability

to readily distribute liquid within itself, and (iii) the ability to remain collapsed but then expand, upon absorption of sufficient liquid, along with the preferred hydrogel-forming absorbent polymers as they swell upon imbibition of liquid. This latter property maintains contact between the foam material and the hydrogel-forming particles as the member  
5 absorbs fluid.

In the present invention, the agglomerate is formed without the use of a separate bonding substance, such as an adhesive. While hydrogel-forming absorbent polymer particles and high surface area open-celled hydrophilic foam particles have previously been combined, the use of an adhesive material has been critical to their combination.  
10 For example, attaching absorbent particles to molten high surface area open-celled hydrophilic material is disclosed in European Patent Publication EP 156,160, in which molten material is extruded so as to produce a stream of melt blown microfibers and, while they are still tacky, absorbent particles are directed therein so they adhere to the fibers.  
15 The technique of entrapment of particulate absorbent materials in a meltblown web is disclosed in U.S. Patent No. 4,923,454, in which microfiber-containing absorbent structures and absorbent articles in which wettable hydrophilic nylon meltblown microfibers and hydrogel-forming polymer particles are disclosed, and in U.S. Patent No. 4,773,903 in which meltblown microfiber and hydrogel-forming polymer particles and  
20 crimped staple fibers and hydrophilizing agent are disclosed. The disclosure of these patents is incorporated by reference herein.

Entrapping high surface area materials and hydrogel-forming absorbent polymer by the techniques generally described in U.S. Patent No. 4,764,325, and U.S. Application Serial No. 09/258,889 both of which are incorporated herein by reference, is  
25 also possible. The high surface area material and hydrogel-forming polymer may also be encapsulated by the techniques generally described in U.S. Patent Application Serial No. 08/585,278, the disclosure of which is incorporated herein by reference.

A unique aspect of the present invention is that the hydrogel-forming absorbent polymer particles and high surface area open-celled hydrophilic foam particles are  
30 formed into an agglomerate without the use of a separate bonding substance, such as an adhesive. The particles of hydrogel-forming absorbent polymer are wetted with water so that they become partially swollen. The amount of water used to wet the absorbent polymer must be limited so that the absorbent polymer particles do not swell appreciably. For purposes of this application, appreciable swelling is defined as swelling that results

in deteriorated performance of the resulting agglomerate (e.g., decreased capillary suction capacity). The partly swollen hydrogel-forming absorbent polymer particles are then mixed and pressed with particles of high surface area open-celled hydrophilic foam. Upon drying, the high surface area open-celled hydrophilic foam particles are effectively  
5 locked into the hydrogel-forming absorbent polymer particles. The physical form of the high surface area open-celled hydrophilic foam material (e.g., particle size) and the hydrogel-forming absorbent polymer (e.g., particle size, form of crosslinking) will dictate, at least to some degree, what processes may be utilized for forming the agglomerate.

In order to form an agglomerate of the hydrogel-forming absorbent polymer  
10 particle and the high surface area open-celled hydrophilic foam particles, pressure must be put upon the mixture of partially swollen hydrogel-forming absorbent polymer particle and foam particles. Such pressure, as can be achieved in a carver or flat press, forces the particles into each other. Pressure may be exerted by any method sufficient to force the particles into each other to a degree where upon drying the agglomerate does not sift  
15 or separate into particles of hydrogel-forming absorbent polymer and high surface area open-celled hydrophilic foam but the method will preferably avoid excessively shearing the partly swollen polymer particle. Excessive shearing can lead to a deterioration in performance properties (e.g., decreased capillary sorption absorbent capacity). Excessive shearing can also be detected by an examination of a SEM micrograph of the  
20 resulting agglomerates, in those agglomerates in which the particles have undergone excessive shearing, swollen hydrogel-forming absorbent polymer particle(s) will be spread across the hydrophilic foam particles, rather than appearing as particles which have merely been pressed into the surface of the hydrophilic foam particles. Another suitable method for forming an agglomerate could include use of a low shear extruder.  
25 After mixing, the material is dried in an oven or other suitable drying apparatus such as a fluid bed dryer.

It is observed that if the hydrogel-forming absorbent polymer particles to be incorporated into the agglomerate are homogeneously cross-linked, substantially less pressure, as compared to the surface cross-linked polymer particles, will be necessary to  
30 achieve an agglomerate. Homogeneously cross-linked hydrogel-forming absorbent polymer particles are formed by incorporating the cross-linking agent during the polymerization of the monomer. Such a hydrogel-forming absorbent polymer particle may be sticky when wetted. With this type of hydrogel-forming absorbent polymer particle, mere mixing and light pressure of the partially swollen polymer particle with the

particles of the high surface area open-celled hydrophilic foam, such as stirring and light pressure in a mortar and pestle bowl, may be sufficient to form an agglomerate. On a commercial scale, for example, a low shear mixer could be utilized.

In another embodiment of the invention, the agglomerate is adjacent to at least one sheet, strip or piece of high surface area open-celled hydrophilic foam material. For example, the agglomerate may be placed upon one sheet of high surface area open-celled hydrophilic foam material, or alternatively, the agglomerate may be positioned between two sheets of high surface area open-celled hydrophilic foam material. In addition, other structures which incorporate the agglomerate of the invention adjacent to at least one sheet, strip or piece of high surface area open-celled hydrophilic foam material are within the scope and spirit of the invention. The agglomerate and at least one foam sheet, strip or piece combination may be incorporated into absorbent storage members in various ways. Figure 6(a) is an illustration of a structure where the agglomerate 100 is positioned between two sheets 102 of high surface area open-celled hydrophilic foam material. Figure 6(b) is an illustration of a structure where the agglomerate 100 is adjacent one sheet 102 of high surface area open-celled hydrophilic foam material. The layered combination of the agglomerate and at least one foam sheet may be manipulated (e.g., cut through its thickness) in a machine and/or the cross direction to cause the sheets to separate into strips or pieces, to provide an storage absorbent member comprised of small sections of the layered combination. Figure 6(c) is an illustration of a structure where the layered combination of the agglomerate and one foam sheet has been cut into strips and stacked together. (It is noted that a structure formed prior to cutting is also useful as a storage absorbent member, and may provide benefits in terms of manufacture on a commercial scale.)

#### 25 D. Absorbent Storage Members

The agglomerates described above may be incorporated into an absorbent storage member. The amount of agglomerate and form of agglomerate (e.g., agglomerate, agglomerate adjacent at least one foam sheet, strip or piece, etc.) incorporated into the storage absorbent member may vary significantly. Furthermore, the concentration of the agglomerate and thus of the hydrogel-forming absorbent polymer and high surface area open-celled hydrophilic foam may vary throughout a given member. In other words, a member may have regions of relatively higher and relatively lower concentrations of agglomerate.

In measuring the concentration in a given region of an absorbent member, the percent by weight of the hydrogel-forming absorbent polymer relative to the combined weight of hydrogel-forming absorbent polymer and any other components (e.g., fibers, polymeric foams, etc.) that are present in the region containing the hydrogel-forming polymer is used. With this in mind, the agglomerate may be present in such an amount that the member will preferably comprise at least about 1%, by weight (on an aggregate basis), high surface area open-celled hydrophilic foam. The member will preferably comprise at least about 10%, by weight, more preferably at least about 15%, by weight, still more preferably at least about 20%, by weight, high surface area open-celled hydrophilic foam. Typically, such member will comprise from about 1 to about 98%, by weight, more typically from about 10 to about 90%, by weight, still more typically from about 15 to about 85%, by weight, still more typically from about 20 to about 80%, and still more typically from about 20 to about 50%, by weight, of the high surface area open-celled hydrophilic foam material. As discussed above, these weight % ranges are based on the aggregate weights of the respective materials in the storage absorbent member; it is recognized that regions of the storage absorbent member may contain greater and lesser amounts of the materials.

#### E. Optional Components and Materials

Storage absorbent members according to the present invention can include other optional components that can be present in absorbent webs. For example, a reinforcing scrim can be positioned within the storage absorbent member, or between the respective absorbent members of the absorbent core. Such reinforcing scrims should be of such configuration as to not form interfacial barriers to liquid transfer, especially if positioned between the respective absorbent members of the absorbent core. In addition, several binders may be used to provide dry and wet integrity to the absorbent core and/or the absorbent storage member itself. In particular, hydrophilic glue fibers may be used to provide bonds between the high surface area materials and the osmotic absorbent material. This is in particular critical for particulate high surface area materials. It is preferred that the amount of binder used is as low as possible, so as not to impair the capillary sorption properties of the absorbent member. However, the skilled artisan will recognize that there are also binders that may enhance the capillary sorption properties of the absorbent member such as fiberized hydrophilic glue with sufficiently high surface area. In this case, the high surface area hydrophilic glue may provide both the liquid handling function and the integrity function, in one material. Also, the respective



absorbent member, or the entire absorbent core, can be enveloped within a liquid pervious sheet, such as a tissue paper sheet or nonwoven web, to obviate user concern regarding loose particulate hydrogel-forming absorbent polymer, as long as the capillary continuity is not disturbed.

- 5           Other optional components that can be included are materials to control odor, contain fecal matter, etc. Also, any absorbent member comprising particulate osmotic absorbent or high surface area material, or the entire absorbent core, can be enveloped within a liquid pervious sheet, such as a tissue paper sheet or nonwoven web, to obviate user concern regarding loose particulate hydrogel-forming absorbent polymer.

10   IV.   Other Storage Absorbent Member Materials and Properties

- The high capillary suction absorbent capacity storage absorbent members of the present invention will comprise the agglomerate comprised of particulate hydrogel-forming absorbent polymer and particulate high surface area open-celled hydrophilic foam. In an alternative embodiment, the high capillary suction storage absorbent members of the present invention comprise the agglomerate positioned adjacent at least one sheet, strip or piece of high surface area open-celled hydrophilic foam. (A non-exhaustive list of optional materials useful in the members is discussed below.) These high suction capacity absorbent members can function as liquid storage members in the absorbent core. The principle function of such liquid storage members is to absorb the discharged body liquid either directly or from other absorbent members (e.g., liquid acquisition/distribution members), and then retain such liquid, even when subjected to pressures normally encountered as a result of the wearer's movements. It should be understood, however, that such absorbent members can serve functions other than liquid storage.

- 25           Other materials generally known in the art may be included in the absorbent storage members, provided they are included at levels sufficiently low so the requisite capillary suction properties are achieved. Optional materials that may also be included in the storage members of the present invention include fibrous materials commonly combined with hydrogel-forming absorbent polymers. For example, wood-based fibers can be included, as can synthetic fibers. However, because such materials will tend to reduce the capillary suction capacity of the storage members comprising the high surface area material and the hydrogel-forming polymer, they will be included at relatively low levels, such that the members still provide the desired capillary suction

absorbent capacity. Indeed, it may be preferred to exclude the use of such fibers, insofar as they add bulk and reduce capillary sorption capacity on a weight basis.

While the basic weight of the storage absorbent members of the present invention is not critical and will vary depending on the end-use of the member (i.e., incorporation into, e.g., a feminine hygiene product, an infant diaper, an adult incontinent product, a bandage), the members will typically have a basis weight of from about 5 to about 3000 g/m<sup>2</sup>, or from about 40 to about 2500 g/m<sup>2</sup>, or from about 100 to about 2000 g/m<sup>2</sup>, or from about 150 to about 1500 g/m<sup>2</sup>, or from about 250 to about 1000 g/m<sup>2</sup>.

#### V. Absorbent Articles

The high suction storage absorbent members of the present invention (comprised of an agglomerate of a particulate hydrogel-forming absorbent polymer and a particulate high surface area open-celled hydrophilic foam in varying forms (e.g., agglomerate, agglomerate adjacent at least one sheet, strip or piece of high surface area open-celled hydrophilic foam)) are particularly useful as the storage portion of the absorbent structures (e.g., absorbent cores or core elements) for various absorbent articles. By "absorbent article" herein is meant a product (suitable for purchase by a consumer) that is capable of absorbing significant quantities of urine, menses, or other fluids (i.e., liquids), such as aqueous fecal matter (runny bowel movements), discharged by an incontinent wearer or user of the article. Examples of such absorbent articles include disposable diapers, incontinence garments, wipes, catamenials such as tampons and sanitary napkins, disposable training pants, bed pads, bandages, and the like. The storage absorbent members herein are particularly suitable for use in articles such as diapers, incontinence pads or garments, wipes, clothing shields, bandages, and the like.

In its simplest form, an absorbent article of the present invention need only include a storage absorbent member of the present invention, but will typically include a backing sheet, typically relatively liquid-impervious, and the high suction storage member. In another simple form, the absorbent article need only include a backing sheet, an acquisition material, and the high suction storage member. The components will be associated such that the acquisition material is positioned so as to acquire the liquid discharge of the wearer of the absorbent article. The high suction member described herein is located so as to be in liquid communication with the acquisition member, or any optional liquid distribution member that is in liquid or capillary communication with the acquisition member. Liquid impervious backing sheets can

comprise any material, for example polyethylene or polypropylene, having a thickness of about 1.5 mils (0.038 mm), which will help retain liquid within the absorbent article.

More conventionally, these absorbent articles will also include a liquid-pervious topsheet element that covers the side of the absorbent article that touches the skin of the wearer. In this configuration, the article includes an absorbent core comprising one or more storage absorbent members of the present invention positioned between the backing sheet and the topsheet. Liquid-pervious topsheets can comprise any material such as polyester, polyolefin, rayon and the like that is substantially porous and permits body liquid to readily pass there through and into the underlying absorbent core. The topsheet material will preferably have no propensity for holding aqueous liquids in the area of contact between the topsheet and the wearer's skin.

In addition to the storage absorbent member of the present invention, the absorbent core of the absorbent articles herein can also comprise other, e.g., conventional, elements or materials. In one embodiment involving a combination of the absorbent member herein and other absorbent materials, the absorbent articles can employ a multi-layer absorbent core configuration where a core layer containing one or more absorbent storage members of the present invention can be used in combination with one or more additional separate core layers comprising other absorbent structures or materials. These other absorbent structures or materials, for example, can include air-laid or wet-laid webs of wood pulp or other cellulosic fibers. These other absorbent structures can also comprise foams, e.g., absorbent foams or even sponges useful as liquid acquisition/distribution components such as those disclosed in U.S. Patent No. 5,563,179 (Stone et al.) issued Oct. 8, 1996, the disclosure of which is incorporated herein by reference.

Another preferred embodiment entails a further separation of the various absorbent core elements. This preferred absorbent core comprises an acquisition layer only around the crotch region of the wearer to manage the initial rapid liquid gush. A distribution layer is positioned vertically to the front and back of the acquisition layer so as to wick the liquid out of the crotch region. The storage layer is positioned in a position near the front and rear waist regions, and is in contact only with the distribution material. The storage absorbent member(s) then must be able to absorb the liquid from the distribution layer, overcoming both the force due to gravity and that due to the desorption pressures of the distribution material. The product so depicted removes liquid from the crotch region within the time provided between insults, leaving the acquisition region

relatively dry and ready for further uptake of liquid. This further maintains the shape of the garment and keeps the crotch area relatively dry for better skin health. See, e.g., co-pending U.S. Patent Application Serial No. 08/825,072, filed March 27, 1997 by G. Young et al., U.S. Patent No. 6,015,935 (G. LaVon et al.), issued January 18, 2000, and  
5 U.S. Patent No. 5,827,253 (G. Young et al.), issued October 27, 1998, which are incorporated by reference herein.

Figure 4 shows a preferred embodiment of a diaper 60 in which the topsheet 61 and the backsheet 62 are co-extensive and have length and width dimensions generally larger than those of the absorbent core 28. The topsheet 61 is joined with and  
10 superimposed on the backsheet 62 thereby forming the periphery of the diaper 60. The periphery defines the outer perimeter or the edges of the diaper 60.

The topsheet 61 is compliant, soft feeling, and non-irritating to the wearer's skin. Further, the topsheet 61 is liquid pervious permitting liquids to readily penetrate through its thickness. A suitable topsheet 61 can be manufactured from a wide range of  
15 materials such as porous foams, reticulated foams, apertured plastic films, natural fibers (e.g., wood or cotton fibers), synthetic fibers (e.g., polyester or polypropylene fibers) or from a combination of natural and synthetic fibers. Typically, the topsheet 61 is made of a hydrophobic material, treated to be initially hydrophilic, to isolate the wearer's skin from liquids in the storage absorbent member 10. The hydrophilic treatment causes initial  
20 wettability of the topsheet so liquid discharges can penetrate the topsheet. A particularly preferred topsheet 61 comprises staple length polypropylene fibers having a denier of about 1.5, such as Hercules type 151 polypropylene marketed by Hercules, Inc. of Wilmington, Delaware. As used herein, the term "staple length fibers" refers to those fibers having a length of at least about 15.9 mm (0.62 inches).

25 There are a number of manufacturing techniques which can be used to manufacture the topsheet 61. For example, the topsheet 61 can be woven, nonwoven, spunbonded, carded, or the like. A preferred topsheet is carded, and thermally bonded by means well known to those skilled in the fabrics art. Preferably, the topsheet 61 has a weight from about 18 to about 25 grams per square meter, a minimum dry tensile  
30 strength of at least about 400 grams per centimeter in the machine direction, and a wet tensile strength of at least about 55 grams per centimeter in the cross-machine direction.

While it is preferred to have a topsheet as the material nearest the wearer's skin, it is not necessary. It is contemplated that a suitable absorbent core configuration could be used without a topsheet and still produce desirable results such as comfort and

absorbency as well as simplicity in manufacturing and material cost savings. For example, the body-side surface of the absorbent core itself could be made of liquid pervious, soft, compliant, non-irritating materials that substitute for a separate topsheet. Such an absorbent core would only need to be used in combination with a backsheet to provide for comfort and absorbency in an absorbent article.

The backsheet 62 is impervious to liquids and is preferably manufactured from a thin plastic film, although other flexible liquid impervious materials may also be used. The backsheet 62 prevents the exudates absorbed and contained in the storage absorbent member 10 from wetting articles which contact the diaper 60 such as bed sheets and undergarments. Preferably, the backsheet 62 is polyethylene film having a thickness from about 0.012 mm (0.5 mil) to about 0.051 centimeters (2.0 mils), although other flexible, liquid impervious materials can be used. As used herein, the term "flexible" refers to materials which are compliant and which will readily conform to the general shape and contours of the wearer's body.

A suitable polyethylene film is manufactured by Monsanto Chemical Corporation and marketed in the trade as Film No. 8020. The backsheet 62 is preferably embossed and/or matte finished to provide a more cloth-like appearance. Further, the backsheet 62 may be "breathable," permitting vapors to escape from the absorbent core 28 while still preventing exudates from passing through the backsheet 62. It is contemplated that a backsheet that is highly breathable but substantially impervious to liquid may be desirable for certain absorbent articles.

The size of the backsheet 62 is dictated by the size of the absorbent core 28 and the exact diaper design selected. In a preferred embodiment, the backsheet 62 has a modified hourglass-shape extending beyond the absorbent core 28 a minimum distance of at least about 1.3 centimeters to at least about 2.5 centimeters (about 0.5 to about 1.0 in.) around the entire diaper periphery.

The topsheet 61 and the backsheet 62 are joined together in any suitable manner. As used herein, the term "joined" encompasses configurations whereby the topsheet 61 is directly joined to the backsheet 62 by affixing the topsheet 61 directly to the backsheet 62, and configurations whereby the topsheet 61 is indirectly joined to the backsheet 62 by affixing the topsheet 61 to intermediate members which in turn are affixed to the backsheet 62. In a preferred embodiment, the topsheet 61 and the backsheet 62 are affixed directly to each other in the diaper periphery by attachment means (not shown) such as an adhesive or any other attachment means as known in the

art. For example, a uniform continuous layer of adhesive, a patterned layer of adhesive, or an array of separate lines or spots of adhesive can be used to affix the topsheet 61 to the backsheet 62.

5 Tape tab fasteners 65 are typically applied to the waistband region 63 of the diaper 60 to provide a fastening means for holding the diaper on the wearer. The tape tab fasteners 65 depicted are representative only. The tape tab fasteners can be any of those well known in the art, such as the fastening tape disclosed in U.S. Patent 3,848,594 (Buell), issued November 19, 1974, which is incorporated by reference. These tape tab fasteners or other diaper fastening means are typically applied near the  
10 corners of the diaper 60.

Elastic members 69 are disposed adjacent the periphery of the diaper 60, preferably along each longitudinal edge 64, so that the elastic members tend to draw and hold the diaper 60 against the legs of the wearer. Additionally, elastic members 67 can be disposed adjacent either or both of the waistband regions 63 of the diaper 60 to  
15 provide a waistband as well as or rather than leg cuffs. For example, a suitable waistband is disclosed in U.S. Patent 4,515,595 (Kievit et al.), issued May 7, 1985, which is incorporated by reference. In addition, a method and apparatus suitable for manufacturing a disposable diaper having elastically contractible elastic members is described in U.S. Patent 4,081,301 (Buell), issued March 28, 1978, which is incorporated  
20 by reference.

The elastic members are secured to the diaper 60 in an elastically contractible condition so that in a normally unrestrained configuration, the elastic members effectively contract or gather the diaper 60. The elastic members can be secured in an elastically contractible condition in at least two ways. For example, the elastic members can be  
25 stretched and secured while the diaper 60 is in an uncontracted condition. Alternatively, the diaper 60 can be contracted, for example, by pleating, and the elastic members secured and connected to the diaper 60 while the elastic members are in their unrelaxed or unstretched condition. The elastic members may extend along a portion of the length of the diaper 60. Alternatively, the elastic members can extend the entire length of the  
30 diaper 60, or any other length suitable to provide an elastically contractible line. The length of the elastic members is dictated by the diaper design.

In use, the diaper 60 is applied to a wearer by positioning one waistband region under the wearer's back, and drawing the remainder of the diaper 60 between the wearer's legs so that the other waistband region is positioned across the front of the

wearer. The tape-tab 65 or other fasteners are then secured preferably to outwardly facing areas of the diaper 60. In use, disposable diapers or other absorbent articles incorporating the storage absorbent members of the present invention tend to more efficiently store liquids and to remain dry due to the high absorbent capacity and high suction capacity of the absorbent members.

When used as an absorbent core in a disposable diaper 60, a preferred embodiment of the core 28 according to the present invention is positioned such that an acquisition strip 52 is in liquid communication with topsheet 61, and serves to quickly acquire and partition body exudates from the wearer's body to an absorptive distribution strip 51. Adhesive bonding of acquisition strip 52 to topsheet 61 may enhance the liquid communication by providing interfacial bonding and preventing topsheet separation from impeding liquid flow. The distribution material 51 moves liquid in the x and y dimensions of the core 28 and is subsequently desorbed by the liquid storage component, shown generally as 10, which is a storage absorbent member of the present invention. While components 52 and 51 are shown generally as being rectilinear and of equal size, other shapes and size relationships may be utilized. As shown, the generally rectilinear components have a width 53 corresponding to a suitable width for the crotch area 66 of a disposable diaper. As well, the length of the respective core components may be varied to provide a suitable fit for various wearer sizes.

As is shown in Figure 4, storage absorbent member 10 can comprise two separate storage absorbent members 20 and 30 such that there is no storage absorbent member element located in the liquid discharge region of the diaper. Because such a storage absorbent member 10 has little or no liquid storage material (it should be recognized that the distribution material 51 may have significant storage capacity and will contain liquid at least until it is desorbed by the higher suction storage material) in the center of the core (corresponding to the crotch or liquid discharge region of the core), articles containing such cores may provide improved fit and wearer comfort both when the article is dry and after it has received several loadings of body liquid. See, e.g., co-pending U.S. Patent Application Serial No. 08/825,072, filed March 27, 1997 by G. Young et al., U.S. Patent No. 6,015,935 (G. LaVon et al.), issued January 18, 2000, and U.S. Patent No. 5,827,253 (G. Young et al.), issued October 27, 1998, Figure 2a depicts a blown-apart view of absorbent core 28 having two separated elements 20 and 30, each of which consist of a storage absorbent member of the present invention. Front panel 20 generally corresponds to the portion of the disposable diaper worn in the front of the

wearer. Similarly, the back panel **30** corresponds to the portion of the disposable diaper worn in the back of the wearer.

Alternatively, storage absorbent member **10** may be a unitary layer(s) (i.e., where the dashed lines 70 in 4 indicate that storage absorbent member **10** is included in the liquid discharge region of the article) of storage absorbent material of the present invention. Such an embodiment of an absorbent core **28** is depicted in Figure 2b.

In one embodiment, acquisition strip **52** will be a liquid handling layer, positioned in the liquid discharge region of the wearer of the article, in the form of a high loft nonwoven, but is preferably in the form of a liquid acquisition layer comprising a layer of modified cellulosic fibers, e.g., stiffened curled cellulosic fibers, and optionally up to about 10% by weight of this liquid acquisition/distribution layer of polymeric gelling agent. In a preferred embodiment, acquisition strip **52** will comprise a high loft chemically bonded polyethylene terephthalate (PET) nonwoven layer (e.g., having a basis weight of about 42 g/m<sup>2</sup>) overlying a layer of stiffened curled cellulosic fibers (e.g., available from Weyerhaeuser Co. WA as CMC®; also available from The Procter & Gamble Co., Paper Technology Division, Cincinnati, OH), such that the PET nonwoven layer is positioned between the stiffened curled cellulosic fibers and the topsheet. The modified cellulosic fibers used in the liquid acquisition layer **52** of such a preferred absorbent article are preferably wood pulp fibers that have been stiffened and curled by means of chemical and/or thermal treatment. Such modified cellulosic fibers are of the same type as are employed in the absorbent articles described in U.S. Patent No. 4,935,022 (Lash et al.), issued June 19, 1990, which is incorporated herein by reference. A preferred embodiment is one where the liquid distribution layer **51** is as described in U.S. Patent No. 6,013,589 (T. A. DesMarais et al.), issued January 11, 2000, U.S. Patent No. 5,800,416 (G. Seger et al.), issued September 1, 1998, each of which is incorporated by reference herein. In a preferred embodiment utilizing the fibrous distribution materials described in U.S. Patent No. 5,800,416, the distribution material is passed through at least two rolls each with circumferential ridges and grooves, which are run at such a close tolerance that the web undergoes permanent deformation. Similar processes have been developed for treating stretch laminate materials and are described in U.S. Patent No. 5,167,897 (Weber) relating to stretch materials. Essentially, this process provides mechanical treatment of the web. This optional liquid distribution layer is typically positioned between the (upper) liquid-handling (e.g., liquid acquisition material) and the (lower) high suction storage absorbent layer and is in liquid



communication therewith. Absorbent articles that can utilize the storage absorbent members of the present invention in a lower liquid storage layer underlying an upper liquid acquisition/distribution layer containing stiffened curled cellulosic fibers are described in greater detail in the U.S. Patent No. 5,147,345 (Young et al.), issued  
5 September 15, 1992.

As referred to herein, "disposable" absorbent articles are those which are intended to be discarded after a single use (i.e., the original absorbent article in its whole is not intended to be laundered or otherwise restored or reused as an absorbent article, although certain materials or all of the absorbent article may be recycled, reused, or  
10 composted). As used herein, the term "diaper" refers to a garment generally worn by infants and incontinent persons that is worn about the lower torso of the wearer. It should be understood, however, that the present invention is also applicable to other absorbent articles such as incontinent briefs, incontinent pads, training pants, diaper inserts, catamenial pads, sanitary napkins, tampons, bandages, facial tissues, paper  
15 towels, and the like.

## VI. Test Methods

### A. Capillary Sorption

#### Purpose

20 The purpose of this test is to measure the capillary sorption absorbent capacity, as a function of height, of the agglomerates of the present invention. Capillary sorption is a fundamental property of any absorbent that governs how liquid is absorbed into the absorbent structure. In the Capillary Sorption experiment, capillary sorption absorbent capacity is measured as a function of fluid pressure due to the height of the sample  
25 relative to the test fluid reservoir.

The method for determining capillary sorption is well recognized. See Burgeni, A.A. and Kapur, C., "Capillary Sorption Equilibria in Fiber Masses," Textile Research Journal, 37 (1967), 356-366; Chatterjee, P.K., Absorbency, Textile Science and Technology 7, Chapter II, pp 29-84, Elsevier Science Publishers B.V, 1985; and U.S.  
30 Patent No. 4,610,678, issued September 9, 1986 to Weisman et al. for a discussion of the method for measuring capillary sorption of absorbent structures. The disclosure of each of these references is incorporated by reference herein.

### Principle

A porous glass frit is connected via an uninterrupted column of fluid to a fluid reservoir on a balance. The sample is maintained under a constant confining weight during the experiment. As the porous structure absorbs fluid upon demand, the weight loss in the balance fluid reservoir is recorded as fluid uptake, adjusted for uptake of the glass frit as a function of height and evaporation. The uptake or capacity at various capillary suction (hydrostatic tensions or heights) is measured. Incremental absorption occurs due to the incremental lowering of the frit (i.e., decreasing capillary suction).

Time is also monitored during the experiment to enable calculation of initial effective uptake rate (g/g/h) at a 200 cm height.

### Reagents

Test Liquid: Synthetic urine is prepared by completely dissolving the following materials in distilled water.

	<u>Compound</u>	<u>F.W.</u>	<u>Concentration (g/L)</u>
15	KCl	74.6	2.0
	Na <sub>2</sub> SO <sub>4</sub>	142	2.0
	(NH <sub>4</sub> )H <sub>2</sub> PO <sub>4</sub>	115	0.85
	(NH <sub>4</sub> ) <sub>2</sub> HPO <sub>4</sub>	132	0.15
	CaCl <sub>2</sub> ·2H <sub>2</sub> O	147	0.25
20	MgCl <sub>2</sub> ·6H <sub>2</sub> O	203	0.5

### General Description of Apparatus Set Up

The Capillary Sorption equipment, depicted generally as **220** in 5A, used for this test is operated under TAPPI conditions (50% RH, 25°C). A test sample is placed on a glass frit shown in 5A as **202** that is connected via a continuous column of test liquid (synthetic urine) to a balance liquid reservoir, shown as **206**, containing test liquid. This reservoir **206** is placed on a balance **207** that is interfaced with a computer (not shown). The balance should be capable of reading to 0.001 g; such a balance is available from Mettler Toledo as PR1203 (Hightstown, NJ). The glass frit **202** is placed on a vertical slide, shown generally in 5A as **201**, to allow vertical movement of the test sample to expose the test sample to varying suction heights. The vertical slide may be a rodless actuator which is attached to a computer to record suction heights and corresponding times for measuring liquid uptake by the test sample. A preferred rodless actuator is available from Industrial Devices (Novato, CA) as item 202X4X34N-1D4B-84-P-C-S-E,

which may be powered by motor drive ZETA 6104-83-135, available from CompuMotor (Rohnert, CA). Where data is measured and sent from actuator **201** and balance **207**, capillary sorption absorbent capacity data may be readily generated for each test sample. Also, computer interface to actuator **201** may allow for controlled vertical  
5 movement of the glass frit **202**. For example, the actuator may be directed to move the glass frit **202** vertically only after "equilibrium" (as defined below) is reached at each suction height.

The bottom of glass frit **202** is connected to Tygon® tubing **203** that connects the frit **202** to three-way drain stopcock **209**. Drain stopcock **209** is connected to liquid  
10 reservoir **205** via glass tubing **204** and stopcock **210**. (The stopcock **209** is open to the drain only during cleaning of the apparatus or air bubble removal.) Glass tubing **211** connects fluid reservoir **205** with balance fluid reservoir **206**, via stopcock **210**. Balance liquid reservoir **206** consists of a lightweight 12 cm diameter glass dish **206A** and cover **206B**. The cover **206B** has a hole through which glass tubing **211** contacts the liquid in  
15 the reservoir **206**. The glass tubing **211** must not contact the cover **206B** or an unstable balance reading will result and the test sample measurement cannot be used.

The glass frit diameter must be sufficient to accommodate the piston/cylinder apparatus, discussed below, for holding the test sample. The glass frit **202** is jacketed to allow for a constant temperature control from a heating bath. The frit is a 350 mL fritted  
20 disc funnel specified as having 4 to 5.5  $\mu\text{m}$  pores, available from Corning Glass Co. (Corning, NY) as #36060-350F. The pores are fine enough to keep the frit surface wetted at capillary suction heights specified (the glass frit does not allow air to enter the continuous column of test liquid below the glass frit).

As indicated, the frit **202** is connected via tubing to fluid reservoir **205** or balance  
25 liquid reservoir **206**, depending on the position of three-way stopcock **210**.

Glass frit **202** is jacketed to accept water from a constant temperature bath. This will ensure that the temperature of the glass frit is kept at a constant temperature of 88°F (31°C) during the testing procedure. As is depicted in 5A, the glass frit **202** is equipped with an inlet port **202A** and outlet port **202B**, which make a closed loop with a circulating  
30 heat bath shown generally as **208**. (The glass jacketing is not depicted in 5A. However, the water introduced to the jacketed glass frit **202** from bath **208** does not contact the test liquid and the test liquid is not circulated through the constant temperature bath.

The water in the constant temperature bath circulates through the jacketed walls of the glass frit 202.)

Reservoir 206 and balance 207 are enclosed in a box to minimize evaporation of test liquid from the balance reservoir and to enhance balance stability during performance of the experiment. This box, shown generally as 212, has a top and walls, where the top has a hole through which tubing 211 is inserted.

The glass frit 202 is shown in more detail in 5B. Figure 5B is a cross-sectional view of the glass frit, shown without inlet port 202A and outlet port 202B. As indicated, the glass frit is a 350 mL fritted disc funnel having specified 4 to 5.5  $\mu\text{m}$  pores. Referring to Figure 5B, the glass frit 202 comprises a cylindrical jacketed funnel designated as 250 and a glass frit disc shown as 260. The glass frit 202 further comprises a cylinder/piston assembly shown generally as 265 (which comprises cylinder 266 and piston 268), which confines the test sample, shown as 270, and provides a small confining pressure to the test sample. To prevent excessive evaporation of test liquid from the glass frit disc 260, a Teflon ring shown as 262 is placed on top of the glass frit disc 260. The Teflon® ring 262 is 0.0127 cm thick (available as sheet stock from McMaster-Carr as # 8569K16 and is cut to size) and is used to cover the frit disc surface outside of the cylinder 266, and thus minimizes evaporation from the glass frit. The ring outer diameter and inner diameter is 7.6 and 6.3 cm, respectively. The inner diameter of the Teflon® ring 262 is about 2 mm less than the outer diameter of cylinder 266. A Viton® O-ring (available from McMaster-Carr as # AS568A-150 and AS568A-151) 264 is placed on top of Teflon® ring 262 to seal the space between the inner wall of cylindrical jacketed funnel 250 and Teflon® ring 262, to further assist in prevention of evaporation. If the O-ring outer diameter exceeds the inner diameter of cylindrical jacketed funnel 250, the O-ring diameter is reduced to fit the funnel as follows: the O-ring is cut open, the necessary amount of O-ring material is cut off, and the O-ring is glued back together such that the O-ring contacts the inner wall of the cylindrical jacketed funnel 250 all around its periphery.

As indicated, a cylinder/piston assembly shown generally in 5B as 265 confines the test sample and provides a small confining pressure to the test sample 270. Referring to Figure 5C, assembly 265 consists of a cylinder 266, a cup-like Teflon® piston indicated by 268 and, when necessary, a weight or weights (not shown) that fits inside piston 268. (Optional weight will be used when necessary to adjust the combined

weight of the piston and the optional weight so a confining pressure of 0.2 psi is attained depending on the test sample's dry diameter. This is discussed below.) The cylinder 266 is Lexan® bar stock and has the following dimensions: an outer diameter of 7.0 cm, an inner diameter of 6.0 cm and a height of 6.0 cm. The Teflon® piston 268 has the following dimensions: an outer diameter that is 0.02 cm less than the inner diameter of cylinder 266. As shown in Figure 5D, the end of the piston 268 that does not contact the test sample is bored to provide a 5.0 cm diameter by about 1.8 cm deep chamber 290 to receive optional weights (dictated by the test sample's actual dry diameter) required to attain a test sample confining pressure of 0.2 psi (1.4 kPa). In other words, the total weight of the piston 268 and any optional weights (not shown in figures) divided by the test sample's actual diameter (when dry) should be such that a confining pressure of 0.2 psi is attained. Cylinder 266 and piston 268 (and optional weights) are equilibrated at 31°C for at least 30 minutes prior to conducting the capillary sorption absorbent capacity measurement.

A non-surfactant treated or incorporated apertured film (14 cm x 14 cm) (not shown) is used to cover the glass frit 202 during Capillary Sorption experiments to minimize air destabilization around the sample. Apertures are large enough to prevent condensation from forming on the underside of the film during the experiment.

#### Test Sample Preparation

The test sample can be obtained by punching out a 5.4 cm diameter circular-shaped structure from a storage absorbent member, using an arch punch. When the member is a component of an absorbent article, other components of the article must be removed prior to testing. In those situations where the member cannot be isolated from other components of the article without significantly altering its structure (e.g., density, relative disposition of the component materials, physical properties of constituent materials, etc.) or where the member is not a component of an absorbent article, the test sample is prepared by combining all the materials that constitute the member such that the combination is representative of the member in question.

The dry weight of the test sample (used below to calculate capillary sorption absorbent capacity) is the weight of the test sample prepared as above under ambient conditions.

#### Experimental Set Up

1. Place a clean, dry glass frit **202** in a funnel holder attached to the vertical slide **201**. Move the funnel holder of the vertical slide such that the glass frit is at the 0 cm height.
2. Set up the apparatus components as shown in 5A, as discussed above.
- 5 3. Place 12 cm diameter balance liquid reservoir **206** on the balance **207**. Place plastic lid **206B** over this balance liquid reservoir **206** and a plastic lid over the balance box **212** each with small holes to allow the glass tubing **211** to fit through. Do not allow the glass tubing to touch the lid **206B** of the balance liquid reservoir or an unstable balance reading will result and the measurement cannot be used.
- 10 4. Stopcock **210** is closed to tubing **204** and opened to glass tubing **211**. Fluid reservoir **205**, previously filled with test fluid, is opened to allow test fluid to enter tubing **211**, to fill balance fluid reservoir **206**.
5. The glass frit **202** is leveled and secured in place. Also, ensure that the glass frit is dry.
- 15 6. Attach the Tygon® tubing **203** to stopcock **209**. (The tubing should be long enough to reach the glass frit **202** at its highest point of 200 cm with no kinks.) Fill this Tygon® tubing with test liquid from liquid reservoir **205**.
7. Attach the Tygon® tubing **203** to the level glass frit **202** and then open stopcock **209** and stopcock **210** leading from fluid reservoir **205** to the glass frit **202**. (Stopcock **210** should be closed to glass tubing **211**.) The test liquid fills the glass frit **202** and removes all trapped air during filling of the level glass frit. Continue to fill until the fluid level exceeds the top of the glass frit disc **260**. Empty the funnel and remove all air bubbles in the tubing and inside the funnel.
- 20 8. Air bubbles may be removed by inverting glass frit **202** and allowing air bubbles to rise and escape through the drain of stopcock **209**. (Air bubbles typically collect on the bottom of the glass frit disc **260**.) Relevel the frit using a small enough level that it will fit inside the jacketed funnel **250** and onto the surface of glass frit disc **260**.
- 25 9. Zero the glass frit with the balance liquid reservoir **206**. To do this, take a piece of Tygon® tubing of sufficient length and fill it with the test liquid. Place one end in the balance liquid reservoir **206** and use the other end to position the glass frit **202**. The test liquid level indicated by the tubing (which is equivalent to the
- 30

balance liquid reservoir level) is 10 mm below the top of the glass frit disc 260. If this is not the case, either adjust the amount of liquid in the reservoir or reset the zero position on the vertical slide 201.

- 5 9. Attach the outlet and inlet ports from the temperature bath 208 via tubing to the inlet and outlet ports 202A and 202B, respectively, of the glass frit. Allow the temperature of the glass frit disc 260 to come to 31°C. This can be measured by partially filling the glass frit with test liquid and measuring its temperature after it has reached equilibrium temperature. The bath will need to be set a bit higher than 31°C to allow for the dissipation of heat during the travel of water from the bath to the glass frit.

- 10 10. The glass frit is equilibrated for 30 minutes.

#### Capillary Sorption Parameters

The following describes a computer program that will determine how long the glass frit remains at each height.

- 15 In the capillary sorption software program, a test sample is at some specified height from the reservoir of fluid. As indicated above, the fluid reservoir is on a balance, such that a computer can read the balance at the end of a known time interval and calculate the flow rate (Delta reading/time interval) between the test sample and reservoir. For purposes of this method, the test sample is considered to be at
- 20 "equilibrium" when the flow rate is less than a specified flow rate for a specified number of consecutive time intervals. It is recognized that for certain material, actual equilibrium may not be reached when the specified "EQUILIBRIUM CONSTANT" is reached. The time interval between readings is 5 seconds.

- 25 The number of readings in the delta table is specified in the capillary sorption menu as "EQUILIBRIUM SAMPLES". The maximum number of deltas is 500. The flow rate constant is specified in the capillary sorption menu as "EQUILIBRIUM CONSTANT".

The Equilibrium Constant is entered in units of grams/sec, ranging from 0.0001 to 100.000.

- 30 The following is a simplified example of the logic. The table shows the balance reading and Delta Flow calculated for each Time Interval.

Equilibrium Samples = 3

Equilibrium Constant = .0015

Time	Balance	Delta
Interval	Value (g)	Flow (g/sec)
0	0	
1	0.090	0.0180
2	0.165	0.0150
3	0.225	0.0120
4	0.270	0.0090
5	0.295	0.0050
6	0.305	0.0020
7	0.312	0.0014
8	0.316	0.0008
9	0.318	0.0004

Delta Table:

Time	0	1	2	3	4	5	6	7	8	9
Delta1	9999	0.0180	0.0180	0.0180	0.0090	0.0090	0.0090	0.0014	0.0014	0.0014
Delta2	9999	9999	0.0150	0.0150	0.0150	0.0050	0.0050	0.0050	0.0008	0.0008
Delta3	9999	9999	9999	0.0120	0.0120	0.0120	0.0020	0.0020	0.0020	0.0004

The equilibrium uptake for the above simplified example is 0.318 gram.

- 5 The following is the code in C language used to determine equilibrium uptake:

```

/*                                takedata.c                                */
int take_data(int equil_samples,double equilibrium_constant)
{
double    delta;
10 static double deltas[500]; /* table to store up to 500 deltas */
double    value;
double    prev_value;
clock_t   next_time;
int        i;
15 for (i=0; i<equil_samples; i++)
    deltas[i] = 9999.; /* initialize all values in the delta table to
9999. gms/sec */
    delta_table_index = 0; /* initialize where in the table to store the
next delta */
20 equilibrium_reached = 0; /* initialize flag to indicate equilibrium has
not been reached */
    next_time = clock(); /* initialize when to take the next reading */

```



```

prev_reading = 0.; /* initialize the value of the previous reading
from the balance */
while (!equilibrium_reached) { /* start of loop for checking for
5   next_time += 5000L; /* calculate when to take next reading */
   while (clock() < next_time); /* wait until 5 seconds has elapsed from
prev reading */
   value = get_balance_reading(); /* read the balance in grams */
10  delta = fabs(prev_value - value) / 5.0; /* calculate absolute value of flow in last
5 seconds */
   prev_value = value; /* store current value for next loop */
   deltas[delta_table_index] = delta; /* store current delta value in the table of
deltas */
   delta_table_index++; /* increment pointer to next position in
15  table */
   if (delta_table_index == equil_samples) /* when the number of deltas = the number of
*/
       delta_table_index = 0; /* equilibrium samples specified, /*
/* reset the pointer to the start of the
20  table. This way */
/* the table always contains the last xx
current samples. */
   equilibrium_reached = 1; /* set the flag to indicate equilibrium is
reached */
25  for (i=0; i < equil_samples; i++) /* check all the values in the delta table
*/
       if (deltas[i] >= equilibrium_constant) /* if any value is > or = to the equilibrium
constant */
           equilibrium_reached = 0; /* set the equilibrium flag to 0 (not at
30  equilibrium) */
   } /* go back to the start of the loop */
}

```

### Capillary Sorption Parameters

Load Description (Confining Pressure): 0.2 psi load

35 Equilibrium Samples (n): 50

Equilibrium Constant: 0.0005 g/sec

Setup Height Value: 100 cm

Finish Height Value: 0 cm

40 Hydrostatic Head Parameters: 200, 180, 160, 140, 120, 100, 90, 80, 70, 60, 50, 45, 40,  
35, 30, 25, 20, 15, 10, 5 and 0 cm.

The capillary sorption procedure is conducted using all the heights specified above, in the order stated, for the measurement of capillary sorption absorbent capacity. Even if it is desired to determine capillary sorption absorbent capacity at a particular height (e.g., 35 cm), the entire series of hydrostatic head parameters must be completed.

in the order specified. Although all these heights are used in performance of the capillary sorption test to generate capillary sorption isotherms for a test sample, the present disclosure describes the storage absorbent members in terms of their absorbent properties at specified heights of 200, 140, 100, 50, 35 and 0 cm.

#### 5 Capillary Sorption Procedure

- 1) Follow the experimental setup procedure.
- 2) Make sure the temperature bath 208 is on and water is circulating through the glass frit 202 and that the glass frit disc 260 temperature is 31°C.
- 3) Position glass frit 202 at 200 cm suction height. Open stopcocks 209 and  
10 210 to connect glass frit 202 with the balance liquid reservoir 206. (Stopcock 210 is closed to liquid reservoir 205.) Glass frit 202 is equilibrated for 30 minutes.
- 4) Input the above capillary sorption parameters into the computer.
- 5) Close stopcocks 209 and 210.
- 6) Move glass frit 202 to the set up height, 100 cm.  
15
- 7) Place Teflon® ring 262 on surface of glass frit disc 260. Put O-ring 264 on Teflon® ring. Place pre-heated cylinder 266 concentrically on the Teflon® ring. Place test sample 270 concentrically in cylinder 266 on glass frit disc 260. Place piston 268 into cylinder 266. Additional  
20 confining weights are placed into piston chamber 290, if required.
- 8) Cover the glass frit 202 with apertured film.
- 9) The balance reading at this point establishes the zero or tare reading.
- 10) Move the glass frit 202 to 200 cm.
- 11) Open stopcocks 209 and 210 (stopcock 210 is closed to fluid reservoir  
25 205) and begin balance and time readings.

#### Glass Frit Correction (blank correct uptake)

Since the glass frit disc 260 is a porous structure, the glass frit (202) capillary sorption absorption uptake (blank correct uptake) must be determined and subtracted to get the true test sample capillary sorption absorption uptake. The glass frit correction is  
30 performed for each new glass frit used. Run the capillary sorption procedure as described above, except without test sample, to obtain the Blank Uptake (g). The elapsed time at each specified height equals the Blank Time (s).

#### Evaporation Loss Correction

- 1) Move the glass frit **202** to 2 cm above zero and let it equilibrate at this height for 30 minutes with open stopcocks **209** and **210** (closed to reservoir **205**).
- 2) Close stopcocks **209** and **210**.
- 5 3) Place Teflon® ring **262** on surface of glass frit disc **260**. Put O-ring **264** on Teflon® ring. Place pre-heated cylinder **266** concentrically on the Teflon® ring. Place piston **268** into cylinder **266**. Place apertured film on glass frit **202**.
- 10 4) Open stopcocks **209** and **210** (closed to reservoir **205**) and record balance reading and time for 3.5 hours. Calculate Sample Evaporation (g/hr) as follows:

$$[\text{balance reading at 1 hr} - \text{balance reading at 3.5 hr}] / 2.5 \text{ hr}$$

Even after taking all the above precautions, some evaporative loss will occur, typically around 0.10 gm/hr for both the test sample and the frit correction. Ideally, the sample evaporation is measured for each newly installed glass frit **202**.

#### Cleaning the Equipment

New Tygon® tubing **203** is used when a glass frit **202** is newly installed. Glass tubing **204** and **211**, fluid reservoir **205**, and balance liquid reservoir **206** are cleaned with 50% Clorox Bleach® in distilled water, followed by distilled water rinse, if microbial contamination is visible.

##### a. Cleaning after each experiment

At the end of each experiment (after the test sample has been removed), the glass frit is forward flushed (i.e., test liquid is introduced into the bottom of the glass frit) with 250 mL test liquid from liquid reservoir **205** to remove residual test sample from the glass frit disc pores. With stopcocks **209** and **210** open to liquid reservoir **205** and closed to balance liquid reservoir **206**, the glass frit is removed from its holder, turned upside down and is rinsed out first with test liquid, followed by rinses with acetone and test liquid. During rinsing, the glass frit must be tilted upside down and rinse fluid is squirted onto the test sample contacting surface of the glass frit disc. After rinsing, the glass frit is forward flushed a second time with 250 ml synthetic urine. Finally, the glass frit is reinstalled in its holder and the frit surface is leveled.

b. Monitoring glass frit performance

Glass frit performance must be monitored after each cleaning procedure and for each newly installed glass frit, with the glass frit set up at 0 cm position. 50 ml of test liquid are poured onto the leveled glass frit disc surface (without Teflon® ring, O-ring and the cylinder/piston components). The time it takes for the test fluid level to drop to 5 mm above the glass frit disc surface is recorded. A periodic cleaning must be performed if this time exceeds 4.5 minutes.

c. Periodic cleaning

Periodically (see monitoring frit performance, above), the glass frits are cleaned thoroughly to prevent clogging. Rinsing fluids are distilled water, acetone, 50% Clorox Bleach® in distilled water (to remove bacterial growth) and test liquid. Cleaning involves removing the glass frit from the holder and disconnecting all tubing. The glass frit is forward flushed (i.e., rinse liquid is introduced into the bottom of the glass frit) with the frit upside down with the appropriate fluids and amounts in the following order:

1. 250 ml distilled water.
2. 100 ml acetone.
3. 250 ml distilled water.
4. 100 ml 50:50 Clorox®/distilled water solution.
5. 250 ml distilled water.
6. 250 ml test fluid.

The cleaning procedure is satisfactory when glass frit performance is within the set criteria of fluid flow (see above) and when no residue is observable on the glass frit disc surface. If cleaning can not be performed successfully, the frit must be replaced.

Calculations

The computer is set up to provide a report consisting of the capillary suction height in cm, time, and the uptake in grams at each specified height. From this data, the capillary suction absorbent capacity, which is corrected for both the frit uptake and the evaporation loss, can be calculated. Also, based on the capillary suction absorbent capacity at 0 cm, the capillary absorption efficiency can be calculated at the specified heights. In addition, the initial effective uptake rate at 200 cm is calculated.

Blank Correct UptakeCapillary Suction Absorbent Capacity ("CSAC")Initial Effective Uptake Rate at 200 cm ("IEUR")

$$\text{IEUR (g/g/hr)} = \frac{\text{CSAC at 200 cm (g/g)}}{\text{Sample Time at 200 cm (s)}}$$

Reporting

A minimum of 2 measurements should be taken for each test sample and the uptake averaged at each height to calculate capillary sorption absorbent capacity for a given storage absorbent member or a given high surface area material.

B. Vertical Hang Sorption Height (VHSH)

The Vertical Hang Sorption Height ("VHSH") test is effected by selecting a strip of foam of suitable length (typically at least 60 cm) with a width of typically about 1 cm. The strip is hung in a chamber thermostatted to 31°C using clips to suspend the strip. The bottom of the strip is immersed in the test fluid, also at 31°C. The test fluid is preferably synthetic urine as described in U.S. Patent No. 5,599,335 (Goldman et al.) issued February 4, 1997, the disclosure of which is incorporated by reference herein. Over time, the test fluid will wick up the strip and reach an equilibrium point where no further wicking occurs. The test fluid may be dyed to facilitate determination of the equilibrium point. Care must be taken to prevent evaporation from the sample, e.g. by encasing it within a glass tube wherein the glass does not touch the sample, and keeping the sample tube suitably capped. The time required to reach equilibrium may vary for the materials of this invention, and range from about 24 to 96 hr., or more. When no perceptible change in the height of the wicking fluid is observed over a 1 hour period, equilibrium is assumed to have been achieved.

The test strip is removed from the test chamber with care to avoid expressing the fluid held therein. The strip is cut into 2.5 cm sections in length and each section is weighed. For convenience, the initial sections below about 50% of the fully expanded height may be cut into sections that are 2 inches (5.1 cm) in length. These weights are divided by the oven dry weight of the foam to compute the capacity (g/g) at the various heights of the foam. A graph such as is depicted in Figure 1 can be developed by charting the capacities vs. the heights at which the sections were taken. The VHSH height at X % is the height in cm where X% of the 0 cm capacity (or FAC) is retained in the foam. A typical value of importance is the VHSH at 90%. In principle, X may be any

value. The most reproducible measure for VHS is achieved at  $X = 90\%$  within the experience of the inventors. It will be obvious to one skilled in the art that this single point value does not fully express the shape of the curve obtained in a plot of capacity vs. height. The single point however serves as a practical point of comparison for the foams of the present invention.

## VII. Representative Examples

### Example 1 - Preparation of High Surface Area Open-Celled Hydrophilic Foam from a HIPE

#### A) HIPE Preparation

Anhydrous calcium chloride (36.32 kg) and potassium persulfate (189 g) are dissolved in 378 liters of water. This provides the water phase stream to be used in a continuous process for forming a HIPE emulsion.

To a monomer combination comprising distilled divinylbenzene (42.4% divinylbenzene and 57.6% ethyl styrene) (2640 g), 2-ethylhexyl acrylate (4400 g), and hexanedioldiacrylate (960 g) is added a diglycerol monooleate emulsifier (480 g), ditallow dimethyl ammonium methyl sulfate (80g), and Tinuvin 765 (20 g). The diglycerol monooleate emulsifier (Grindsted Products; Brabrand, Denmark) comprises approximately 81% diglycerol monooleate, 1% other diglycerol monoesters, 3% polyols, and 15% other polyglycerol esters, imparts a minimum oil/water interfacial tension value of approximately 2.7 dyne/cm and has an oil/water critical aggregation concentration of approximately 2.8 wt%. After mixing, this combination of materials is allowed to settle overnight. No visible residue is formed and all of the mixture is withdrawn and used as the oil phase in a continuous process for forming a HIPE emulsion.

Separate streams of the oil phase (25°C) and water phase (53°-55°C) are fed to a dynamic mixing apparatus. Thorough mixing of the combined streams in the dynamic mixing apparatus is achieved by means of a pin impeller. The pin impeller comprises a cylindrical shaft of about 36.5 cm in length with a diameter of about 2.9 cm. The shaft holds 6 rows of pins, 3 rows having 33 pins and 3 rows having 34 pins, each of the three pins at each level disposed at an angle of 120° to each other, with the next level down disposed at 60° to its neighboring level with each level separated by 0.03 mm, each having a diameter of 0.5 cm extending outwardly from the central axis of the shaft to a length of 2.3 cm. The pin impeller is mounted in a cylindrical sleeve which forms the dynamic mixing apparatus, and the pins have a clearance of 1.5 mm from the walls of the cylindrical sleeve.

A minor portion of the effluent exiting the dynamic mixing apparatus is withdrawn and enters a recirculation zone, as shown in the Figure of U.S. Patent No. 5,827,909 (DesMarais), issued October 27, 1998, the disclosure of which is incorporated by reference herein. The Waukesha pump in the recirculation zone returns the minor  
5 portion to the entry point of the oil and water phase flow streams to the dynamic mixing zone.

The static mixer (TAH Industries Model 100-812) has 12 elements with a 1 in. (2.5 cm) outside diameter. A hose is mounted downstream from the static mixer to facilitate delivery of the emulsion to the device used for curing. Optionally an additional  
10 static mixer is used to provide addition back pressure to keep the hose filled. The optional static mixer can be a 1 in. (2.5 cm) pipe, 12 element mixer (McMaster-Carr, Aurora, OH, Model 3529K53).

The combined mixing and recirculation apparatus set-up is filled with oil phase and water phase at a ratio of 4 parts water to 1 part oil. The dynamic mixing apparatus  
15 is vented to allow air to escape while filling the apparatus completely. The flow rates during filling are 7.57 g/sec oil phase and 30.3 cc/sec water phase.

Once the apparatus set-up is filled, agitation is begun in the dynamic mixer, with the impeller turning at 1750 RPM and recirculation is begun at a rate of about 30 cc/sec. The flow rate of the water phase is then steadily increased to a rate of 151.3 cc/sec over  
20 a time period of about 1 min., and the oil phase flow rate is reduced to 3.03 g/sec over a time period of about 3 min. The recirculation rate is steadily increased to about 150 cc/sec during the latter time period. The back pressure created by the dynamic zone and static mixers at this point is about 19.9 PSI (137 kPa), which represents the total pressure drop of the system. The Waukesha pump (Model 30) speed is then steadily  
25 decreased to a yield a recirculation rate of about 75 cc/sec.

#### B) Polymerization of HIPE

The HIPE flowing from the static mixer at this point is collected in a round polyethylene tub, 40 in. (102 cm) in diameter and 12.5 in. (31.8 cm) high, with removable sides, much like a springform pan used in cooking cakes. A pipe-like polyethylene insert  
30 12.5 in. (31.8 cm) in diameter at its base is firmly affixed to the center of the base and is 12.5 in. (31.8 cm) high. The HIPE-containing tubs are kept in a room maintained at 65°C for 18 hours to effect polymerization and form the foam.

#### C) Foam Washing and Dewatering

The cured HIPE foam is removed from the curing tubs. The foam at this point has residual water phase (containing dissolved emulsifiers, electrolyte, initiator residues, and initiator) about 48-52 times (48-52X) the weight of polymerized monomers. The foam is sliced with a sharp reciprocating saw blade into sheets which are 0.185 inches (4.7 mm) in thickness. These sheets are then subjected to compression in a series of 2 porous nip rolls equipped with vacuum which gradually reduce the residual water phase content of the foam to about 6 times (6X) the weight of the polymerized material. At this point, the sheets are then resaturated with a 1.5%  $\text{CaCl}_2$  solution at 60°C., are squeezed in a series of 3 porous nip rolls equipped with vacuum to a water phase content of about 4X. The  $\text{CaCl}_2$  content of the foam is between 8 and 10%.

The foam remains compressed after the final nip at a thickness of about 0.021 in. (0.053 cm). The foam is then dried in air for about 16 hours. Such drying reduces the moisture content to about 9-17% by weight of polymerized material. At this point, the foam sheets are very drapeable and "thin-after-drying". The foam sheet may be particularized to form high surface area open-celled hydrophilic foam particles for incorporation into the agglomerate of the invention. The foam sheets so formed may also be utilized in sheet form for the embodiment of the invention wherein the agglomerate is positioned adjacent to at least one strip, sheet or piece of high surface area open-celled hydrophilic foam material.

#### Example 2 - Preparation of High Surface Area Open-Celled Hydrophilic Foam from a HIPE

##### A) HIPE Preparation

The water and oil phase streams to be used in a continuous process for forming a HIPE emulsion is prepared according to Example 1. Separate streams of the oil phase (25°C) and water phase (53°-55°C) are fed to a dynamic mixing apparatus as detailed in Example 1.

Once the apparatus set-up is filled, agitation is begun in the dynamic mixer, with the impeller turning at 1700 RPM and recirculation is begun at a rate of about 30 cc/sec. The flow rate of the water phase is then steadily increased to a rate of 151.3 cc/sec over a time period of about 1 min., and the oil phase flow rate is reduced to 3.36 g/sec over a time period of about 3 min. The recirculation rate is steadily increased to about 150 cc/sec during the latter time period. The back pressure created by the dynamic zone and static mixers at this point is about 19.7 PSI (136 kPa), which represents the total



pressure drop of the system. The Waukesha pump speed is then steadily decreased to a yield a recirculation rate of about 75 cc/sec.

B) Polymerization of HIPE

5 The HIPE flowing from the static mixer at this point is collected and cured into a high surface area open-celled hydrophilic foam as detailed in Example 1.

C) Foam Washing and Dewatering

10 The cured HIPE foam is removed from the curing tubs. The foam at this point has residual water phase (containing dissolved emulsifiers, electrolyte, initiator residues, and initiator) about 43-47 times (43-47X) the weight of polymerized monomers. The foam is sliced with a sharp reciprocating saw blade into sheets which are 0.185 inches (4.7 mm) in thickness. These sheets are then subjected to compression in a series of 2 porous nip rolls equipped with vacuum which gradually reduce the residual water phase content of the foam to about 6 times (6X) the weight of the polymerized material. At this point, the sheets are then resaturated with a 1.5%  $\text{CaCl}_2$  solution at 60°C., are squeezed in a series of 3 porous nip rolls equipped with vacuum to a water phase content of about 4X. The  $\text{CaCl}_2$  content of the foam is between 8 and 10 %.

20 The foam remains compressed after the final nip at a thickness of about 0.028 in. (0.071 cm). The foam is then dried in air for about 16 hours. Such drying reduces the moisture content to about 9-17% by weight of polymerized material. At this point, the foam sheets are very drapeable and "thin-after-drying". As described in Example 1 the high surface area open-celled hydrophilic foam may be particularized or utilized in sheet form.

Example 3 - Preparation of High Surface Area Open-Celled Hydrophilic Foam from a HIPE

25 A) HIPE Preparation

The water and oil phase streams to be used in a continuous process for forming a HIPE emulsion is prepared according to Example 1. Separate streams of the oil phase (25°C) and water phase (53°-55°C) are fed to a dynamic mixing apparatus as detailed in Example 1.

30 Once the apparatus set-up is filled, agitation is begun in the dynamic mixer, with the impeller turning at 1750 RPM and recirculation is begun at a rate of about 30 cc/sec. The flow rate of the water phase is then steadily increased to a rate of 151.3 cc/sec over a time period of about 1 min., and the oil phase flow rate is reduced to 3.78 g/sec over a

time period of about 3 min. The recirculation rate is steadily increased to about 150 cc/sec during the latter time period. The back pressure created by the dynamic zone and static mixers at this point is about 18.7 PSI (129 kPa), which represents the total pressure drop of the system. The Waukesha pump speed is then steadily decreased to a yield a recirculation rate of about 75 cc/sec.

B) Polymerization of HIPE

The HIPE flowing from the static mixer at this point is collected and cured into a high surface area open-celled hydrophilic foam as detailed in Example 1.

C) Foam Washing and Dewatering

The cured HIPE foam is removed from the curing tubs. The foam at this point has residual water phase (containing dissolved emulsifiers, electrolyte, initiator residues, and initiator) about 38-42 times (38-42X) the weight of polymerized monomers. The foam is sliced with a sharp reciprocating saw blade into sheets which are 0.185 inches (4.7 mm) in thickness. These sheets are then subjected to compression in a series of 2 porous nip rolls equipped with vacuum which gradually reduce the residual water phase content of the foam to about 6 times (6X) the weight of the polymerized material. At this point, the sheets are then resaturated with a 1.5%  $\text{CaCl}_2$  solution at 60°C., are squeezed in a series of 3 porous nip rolls equipped with vacuum to a water phase content of about 4X. The  $\text{CaCl}_2$  content of the foam is between 8 and 10%.

The foam remains compressed after the final nip at a thickness of about 0.028 in. (0.071 cm). The foam is then dried in air for about 16 hours. Such drying reduces the moisture content to about 9-17% by weight of polymerized material. At this point, the foam sheets are very drapeable and "thin-after-drying". As described in Example 1 the high surface area open-celled hydrophilic foam may be particularized or utilized in sheet form.

Example 4 - Making Particulate High Surface Area Open-Celled Hydrophilic Foam

This example describes a method for particularizing a high surface area open-celled hydrophilic foam material. 10 g of air dried high surface are open-celled hydrophilic foam (prepared according to Example 2 or in accordance with Example 3 above) is placed in a blender (Osterizer model 848-36L) equipped with a 1.25 liter jar, into which 1 liter of 2% calcium chloride solution has been placed. After ensuring that all of the foam material is submerged, the blender is agitated on the "Liquefy" (high setting) for 10 seconds and then additionally agitated on the "Grate" setting for 5 sec. The resultant slurry is then transferred to a Buchner funnel (Coors USA model 60283) lined

with a paper towel. Approximately 500 ml of fluid is freely drained from the sample. The sample is then covered with a rubber membrane and vacuum is applied (approximately 500 mm Hg) to dewater the sample to a weight of 50 to 60 grams.

5 The sample is returned to a dry blender jar and dispersed with the agitation set on "Liquefy" while the jar and base are inverted and returned to upright several times to disperse the sample to approximately individual particles. The dispersed sample is then air dried under ambient conditions. The particles of foam are then sieved. A particle size fraction is collected which passes through a U.S.A. Series Standard 35 mesh sieve, i.e., a fraction with particles equal to or less than approximately 500 microns. The  
10 sieved high surface area open-celled hydrophilic foam particles may be combined with hydrogel-forming absorbent polymer to form an agglomerate.

Example 5 - Preparation of Mixed-Bed Ion-Exchange Hydrogel-Forming Absorbent

Polymer

(i) Cation-Exchange Absorbent Polymer - Preparation of Crosslinked Polyacrylic  
15 acid

A homogeneously crosslinked polyacrylic acid is synthesized by placing 450 grams of acrylic acid monomer (Aldrich Chemical Co., catalog number 14,723-0; lot number 15930CS) in a clean 4000 mL resin kettle. 7.2 grams of N,N'-methylenebisacrylamide (Aldrich Chemical Co., catalog number 14,607-2; lot number  
20 04511DR) and 0.85 grams of 2,2'-Azobis(2-amidinopropane) dihydrochloride (Wako, lot number P2197) are dissolved in 2050 grams of water and added to the acrylic acid monomer in the resin kettle. The solution is sparged with nitrogen for 15 minutes to remove dissolved oxygen. The resin kettle is then sealed and the solution is heated at 40°C for 16 hours.

25 The resultant gel is allowed to cool and then broken into pieces approximately 1 cm in diameter and dried in a vacuum oven at 55°C for 60 hours. The sample is ground and sieved through a U.S.A. 20 mesh sieve using a Wiley Mill to obtain homogeneously crosslinked polyacrylic acid.

(ii) Anion-Exchange Absorbent Polymer - Preparation of Crosslinked  
30 Polyallylamine

Polyallylamine, 1250 grams of 20% solution (Nitto Boseki Co., LTD, Tokyo, Japan, lot number 80728) is weighed in a 2000 mL glass jar. Ethylene glycol diglycidyl ether, 19 grams of 50% solution (Aldrich Chemical Co., catalog number, E2,720-3) is diluted with 20 grams of distilled water and added to the polyallylamine solution. The

mixture is stirred at room temperature for approximately two minutes before being placed in a vented oven at approximately 60°C overnight.

The resultant gel is broken into pieces approximately 5 mm in diameter and dried under high vacuum for approximately 96 hours to yield a lightly crosslinked polyallylamine anion-exchange absorbent polymer which is stored under a dry atmosphere.

(iii) Mixed-Bed Ion-Exchange Hydrogel-Forming Absorbent Polymer

The crosslinked polyallylamine anion-exchange absorbent polymer is ground and sieved. A particle size fraction is collected which passes through a U.S.A. Series Standard 25 mesh sieve, but not through a U.S.A. Series Standard 70 mesh sieve (i.e., a fraction with particles in the range of approximately 200 to 700 microns in diameter).

Approximately 125 grams of the sieved crosslinked polyacrylic acid (200 to 700 microns diameter) cation-exchange absorbent polymer and 125 grams of the sieved crosslinked polyallylamine anion-exchange absorbent polymer are mixed together so as to distribute the particles of each type of polymer evenly throughout the mixture. This mixture comprises a mixed-bed ion-exchange absorbent polymer composition used in the storage absorbent member of the present Example. Additional information on such compositions is disclosed in U.S. Patent Application Serial No. 09/258,890, filed March 1, 1999 by Hird, et al. (titled "Absorbent Polymer Compositions Having High Sorption Capacities Under an Applied Pressure"), which was previously incorporated herein by reference.

Example 6 - Preparation of Homogeneously Crosslinked Hydrogel-Forming Absorbent Polymer Particles

A homogeneously crosslinked polyacrylic acid is synthesized by placing 480 grams of acrylic acid monomer (Aldrich Chemical Co., catalog number 14,723-0; lot number 15930CS) in a clean 3000 mL resin kettle. Approximately, 1.02 grams of N,N'-methylenebisacrylamide (Aldrich Chemical Co., catalog number 14,607-2; lot number 04511DR) and 0.90 grams of 2,2'-Azobis(2-amidinopropane) dihydrochloride (Wako, lot number P2197) are dissolved in about 1920 grams of deionized water and added to the acrylic acid monomer in the resin kettle. The solution is sparged with nitrogen for 15 minutes to remove dissolved oxygen. The resin kettle is then sealed and the solution is heated at 60°C for 16 hours to yield a homogeneously crosslinked polyacrylic acid gel.

The resultant gel is broken into pieces approximately 5 mm in diameter and placed in a 4000 mL beaker. Approximately 1000 mL of 5N sodium hydroxide solution

(Mallinkrodt, lot number H36922506) and 1000 mL of deionized water are added to the 4000 mL beaker containing the gel pieces. The gel is allowed to absorb the fluid for 16 hours. The resultant gel is dried under high vacuum for approximately 96 hours to yield a homogeneously crosslinked sodium poly acrylate absorbent polymer which is stored  
5 under a dry atmosphere (HGS-AGM1).

The HGS-AGM1 is ground and sieved. A particle size fraction is collected which passes through a U.S.A. Series Standard 25 mesh sieve, but not through a U.S.A. Series Standard 70 mesh sieve (i.e., a fraction with particles in the range of approximately 200 to 700 microns in diameter).

10 **Example 7 – Making Agglomerate Using Homogeneously Crosslinked Hydrogel-Forming Absorbent Polymer Particles**

Dry high surface area open-celled hydrophilic foam particles and hydrogel-forming absorbent polymer particles are weighed into a tared dish measuring at least 8"X8". A metal spatula is used to thoroughly mix together the dry materials and then  
15 spread the mixture evenly over the bottom of the dish. The dish containing the sample is re-tared.

A spray bottle (Arrow Spray King (model #00158), Arrow Manufacturing Company, Elk Grove, IL) set on "spray" is used to lightly mist the sample with distilled water. The sample is then mixed with the spatula. The amount of water that has been  
20 added may be checked by weighing. Misting and mixing of the sample is continued until a total of about 0.6 grams of water (per gram of hydrogel-forming absorbent polymer material (dry weight basis)) has been added. For 15g of polymer, this will equal approximately 10 full sprays. Thoroughly mix the sample using the spatula to evenly distribute the moisture.

25 The sample is transferred to a Coors mortar (#60328, 1900 mL capacity, 210 mm diameter, 130 mm high), and mildly ground with the Coors pestle (#60329) for about 1 minute. The sample is transferred into 2 large weigh boats and lab dried/equilibrated (at 72°F, 50% relative humidity (RH)) for approximately 48 hours.

Two agglomerates were made in accordance with this procedure. Agglomerate A  
30 utilized a 40:60 particulate high surface area open-celled hydrophilic foam to hydrogel-forming absorbent polymer particle ratio, and comprised a mixed bed ion exchange hydrogel-forming absorbent polymer made in accordance with example 5 herein. Agglomerate B also utilized a 40:60 particulate high surface area open-celled hydrophilic foam to hydrogel-forming absorbent polymer particle ratio, but comprised particulate

homogeneously crosslinked hydrogel-forming absorbent polymer made in accordance with example 6 herein. Both agglomerate A and agglomerate B were made with particulate high surface area open-celled hydrophilic foam as set forth in Example 4.

Example 8 – Making Agglomerate Using Surface Crosslinked Hydrogel-forming

5                   Absorbent Polymer)

To construct an agglomerate comprising surface crosslinked hydrogel-forming absorbent polymer particles (such as ASAP 2300 (lot # 470131, available from Chemdal Corporation of Palantine, IL; also available from The Procter & Gamble Co., Paper Technology Division, Cincinnati, OH) and high surface area open-celled hydrophilic foam particles the following method was utilized. Approximately 6.0 grams of surface crosslinked hydrogel-forming absorbent polymer are weighed into a tared 100 mL glass beaker. Approximately 30.0 grams of distilled water (about 5 grams of water per gram of hydrogel-forming absorbent polymer particles, dry weight basis) are added to the glass beaker and the mixture is stirred with a metal spatula. The beaker is covered with  
15                   parafilm and the mixture is allowed to sit for about 15 minutes, during which time the particles of hydrogel-forming absorbent polymer swell).

Approximately 4.0 grams of high surface area open-celled hydrophilic foam particles (made according to Example 4 herein, with a particle size of less than about 500 microns, and a 5.5%  $\text{CaCl}_2$  (dry weight basis) as a hydratable salt) are weighed into  
20                   a large weigh boat. The swollen hydrogel-forming absorbent polymer particles are combined with the high surface area open-celled hydrophilic foam particles and mixed with a metal spatula. The mixture of hydrogel-forming absorbent polymer particles and high surface area open-celled hydrophilic foam particles is transferred to a stainless steel cup (approximately 9.1 cm inner diameter, 11.5 cm outer diameter, 3.2 cm cup depth,  
25                   1.2 cm base thickness). A stainless steel piston (9 cm diameter x 5 cm thickness) is placed into the cup on top of the mixture.

The cup and piston are placed in a Carver Press (Model C), and 150 psi of pressure is applied for approximately 1 minute. The sample is then transferred to a large weigh boat and a metal spatula is used to break large chunks of agglomerate into pieces  
30                   less than or equal to about 3 mm in size. The resulting agglomerate is allowed to lab dry/equilibrate (72° F, 50% relative humidity) for approximately 48 hours.

Example 9 – Making Agglomerate Using Favor 1180 (a homogeneously crosslinked hydrogel-forming absorbent polymer)

To construct an agglomerate comprising high surface area open-celled

hydrophilic foam particles and Favor 1180 as the hydrogel-forming absorbent polymer particles, the method set forth in Example 7 is followed, utilizing Favor 1180 particles (a homogeneously crosslinked polymer, available from Stockhausen Louisiana LLC., Favor 1180; lot no. SLGLG9H422; similar samples of Favor 1180 available from The Procter & Gamble Co., Paper Technology Division, Cincinnati, OH) as the hydrogel-forming absorbent polymer particles.

Example 10 - Preparation of Homogeneously Crosslinked Hydrogel-Forming Absorbent Polymer Particles

A homogeneously crosslinked polyacrylic acid is synthesized by placing 200 grams of acrylic acid monomer (Aldrich Chemical Co., catalog number 14,723-0; lot number 15930CS) in a clean 1000 mL resin kettle. Approximately, 1.07 grams of N,N'-methylenebisacrylamide (Aldrich Chemical Co., catalog number 14,607-2; lot number 04511DR) and 0.37 grams of 2,2'-Azobis(2-amidinopropane) dihydrochloride (Wako, lot number P2197) are dissolved in about 800 grams of deionized water and added to the acrylic acid monomer in the resin kettle. The solution is sparged with nitrogen for 15 minutes to remove dissolved oxygen. The resin kettle is then sealed and the solution is heated to approximately 45°C for 16 hours to yield a crosslinked polyacrylic acid gel. The resultant gel is broken into pieces approximately 5 mm in diameter and dried under high vacuum for approximately 96 hours to yield a homogeneously crosslinked polyacrylic acid which is stored under a dry atmosphere.

To prepare a homogeneously crosslinked sodium poly acrylate absorbent polymer, 24.3 mL of 4N sodium hydroxide solution (J.T. Baker, lot number 16500) and 75 mL of deionized water are added to a 150 mL beaker. Approximately, 10 grams of crosslinked polyacrylic acid is added to the solution and is allowed to absorb the fluid for 3 hours. The resultant gel dried under high vacuum for approximately 96 hours to yield a homogeneously crosslinked sodium poly acrylate which is stored under a dry atmosphere (HGS-AGM2).

The HGS-AGM2 is ground and sieved. A particle size fraction is collected which passes through a U.S.A. Series Standard 25 mesh sieve, but not through a U.S.A. Series Standard 70 mesh sieve (i.e., a fraction with particles in the range of approximately 200 to 700 microns in diameter).

Example 11 - Making Agglomerate Using Homogeneously Crosslinked Hydrogel-Forming Absorbent Polymer Particles

To construct an agglomerate comprising unsifted particulate high surface area

open-celled hydrophilic foam (made in accordance with Example 4 herein, except that the particles are not sieved), approximately 1.83 grams of dry open-celled hydrophilic foam particles are placed into a 100 ml glass beaker. About 13.8 grams of distilled water are added and the particles and distilled water are mixed thoroughly with a metal spatula. The beaker is sealed with parafilm and left to equilibrate for about 2 hours at 72°F.

Approximately 2.75 grams of HGS-AGM2 (as described in Example 10) are added to the wetted open-celled hydrophilic foam particles. The sample is mixed thoroughly using a metal spatula. The beaker is sealed with parafilm and left to equilibrate for about 3 hours at 72°F.

The mixture is extruded on low speed (setting "stir" or "1") through a plate with eight 8.5 mm diameter holes using a KitchenAid Proline Mixer (model # KSM5) with food grinder attachment FGA. The sample is transferred to a 5.5" x 5.5" weigh boat and lab dried/ equilibrated (at 72°F, 50% relative humidity (RH)) for approximately 48 hours.

The resulting agglomerate utilizes a 40:60 particulate high surface area open-celled hydrophilic foam to hydrogel-forming absorbent polymer particle ratio.



**WHAT IS CLAIMED IS:**

1. A high capillary suction storage absorbent member characterized in that said member comprises an agglomerate of particulate hydrogel-forming absorbent polymer and particulate high surface area open-celled hydrophilic foam.
2. The storage absorbent member of Claim 1 wherein said foam is thin-until-wet.
3. The storage absorbent member of Claims 1 or 2 wherein said foam comprises from about 1% to about 98%, by total weight of said member, preferably said foam comprises from about 15% to about 85%, by total weight of said member.
4. A storage absorbent member according to any of the above claims wherein said storage absorbent member has one or more of the following:
  - a) a capillary sorption absorbent capacity at a height of 100 cm of at least about 4 g/g, preferably between about 4 g/g and about 30 g/g, more preferably between about 8 g/g and about 20 g/g;
  - b) a capillary sorption absorbent capacity at a height of 140 cm of at least about 4 g/g; or
  - c) a capillary sorption absorbent capacity at a height of 200 cm of at least about 3 g/g.
5. The storage absorbent member of any of the above claims having a capillary sorption adsorbent capacity at a height of 0 cm of at least about 15 g/g.
6. A storage absorbent member according to Claims 1-3, said storage absorbent member having a capillary sorption absorbent capacity at a height of 0 cm of at least about 15 g/g and having one or more of the following:
  - a) a capillary absorption efficiency at a height of 100 cm of at least about 25%;
  - b) a capillary absorption efficiency at a height of 50 cm of at least about 30%;
  - c) a capillary absorption efficiency at a height of 35 cm of at least about 50%.
7. The agglomerate of any of the above claims wherein said foam comprises particles with a dry particle size of less than about 1000 microns.
8. The agglomerate of any of the above claims having an initial effective uptake rate at 200 cm of at least about 3 g/g/hr.
9. The storage absorbent member according to any of the above claims wherein said member further comprises at least one sheet, strip or piece of high surface area open-celled hydrophilic foam lying adjacent said agglomerate.

10. The storage absorbent member of any of the above claims further comprising an absorbent article selected from the group consisting of a diaper, a catamenial, and a wipe.

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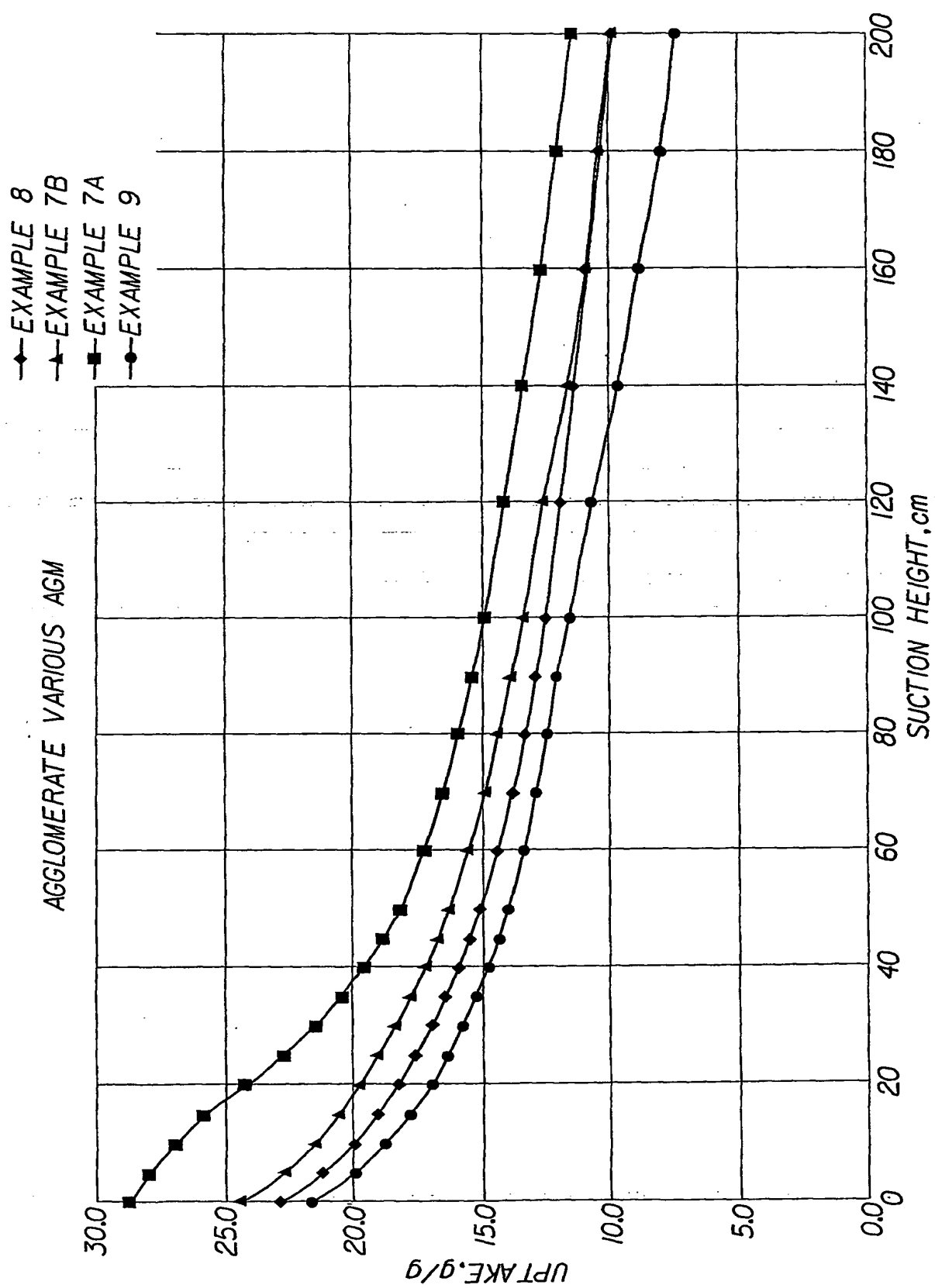


Fig. 1

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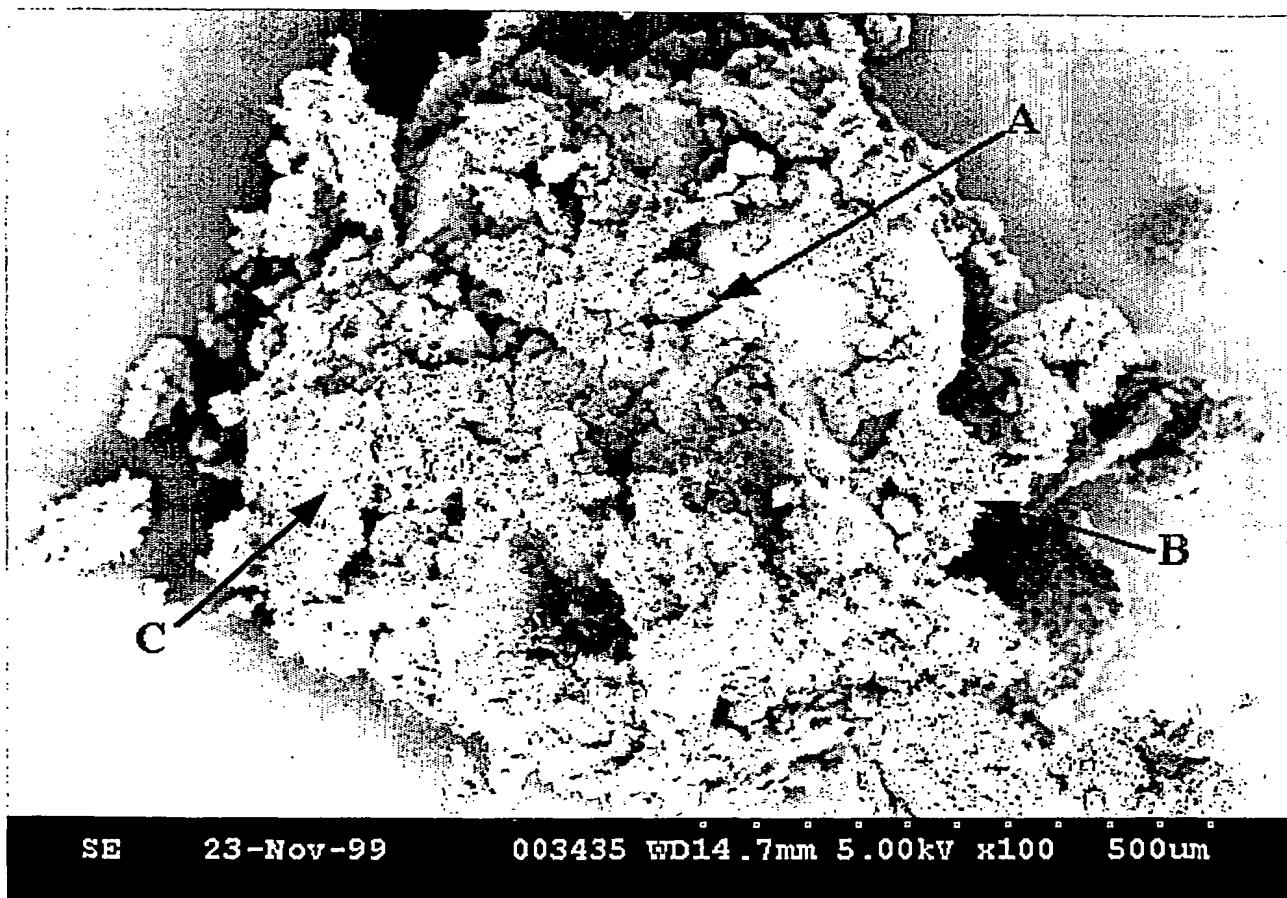


Fig. 2

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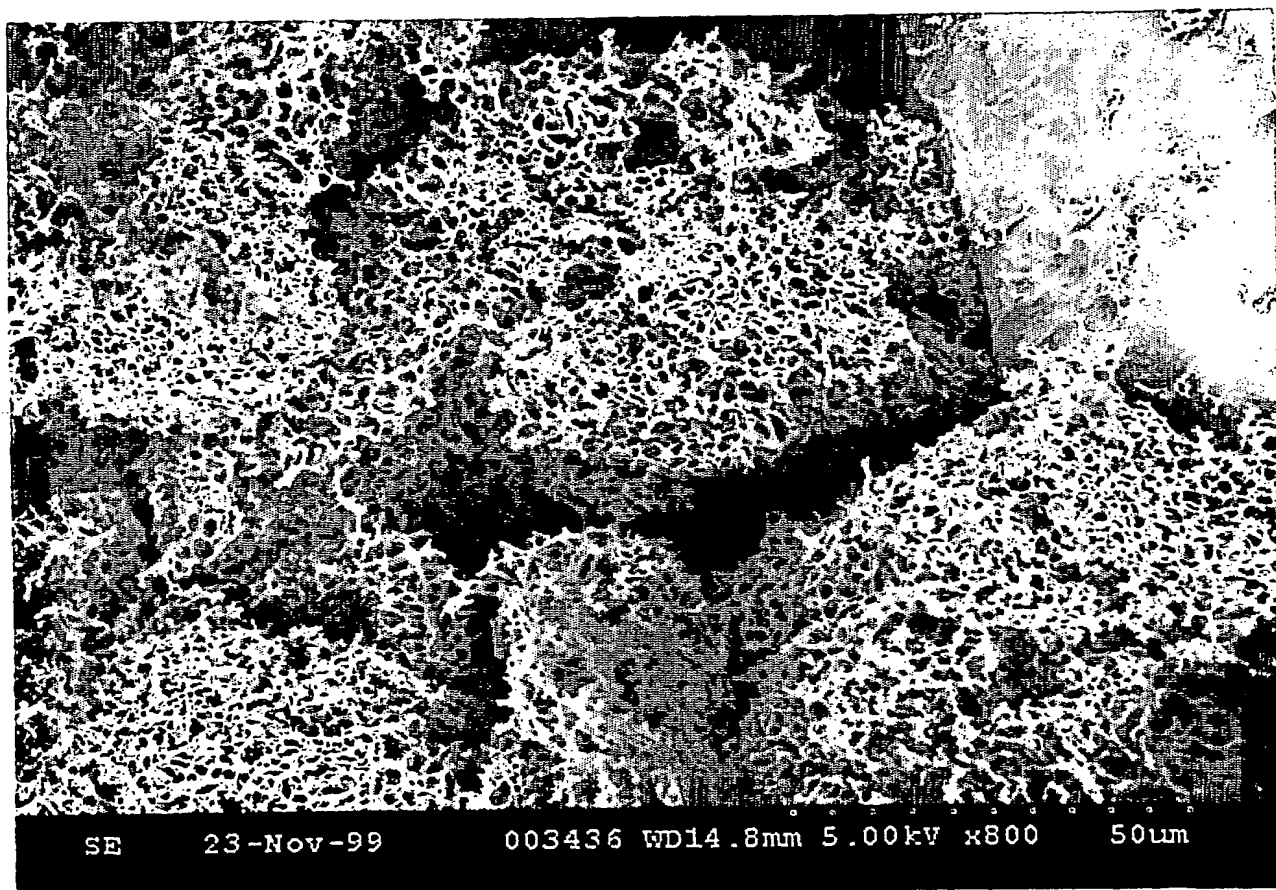


Fig. 3

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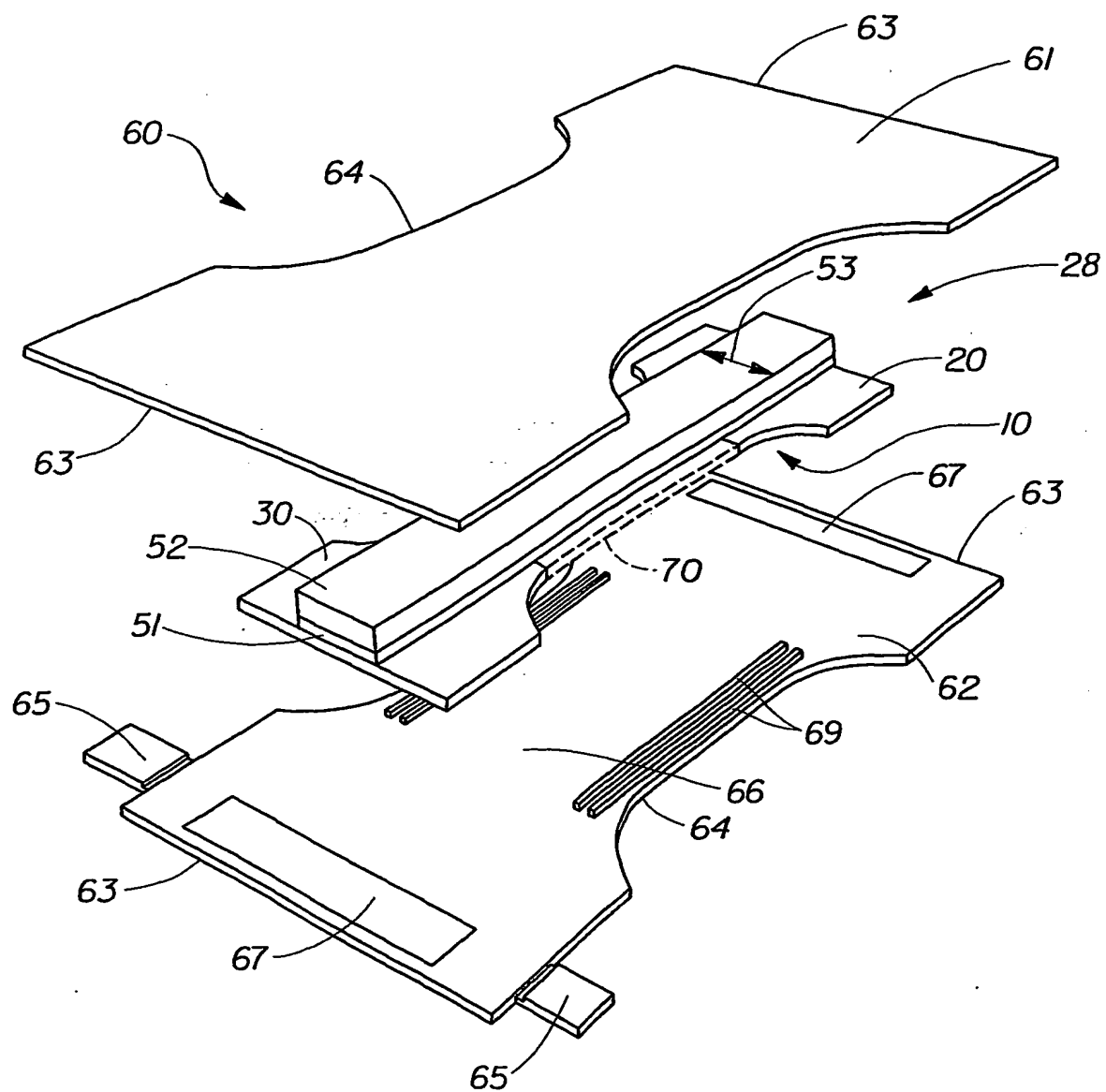


Fig. 4

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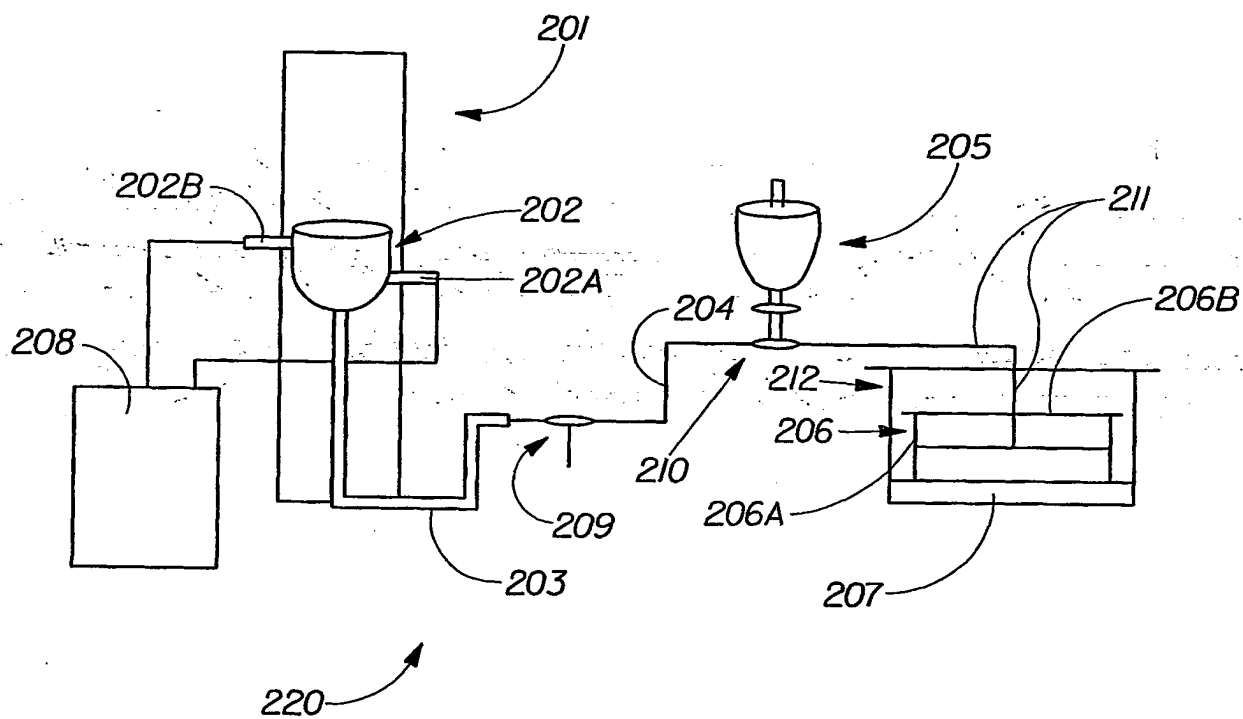
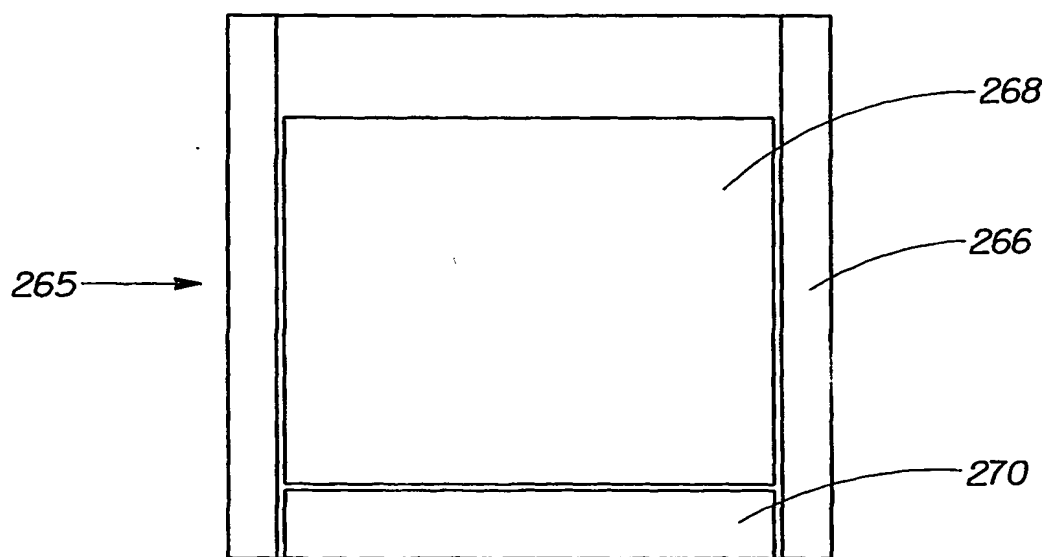
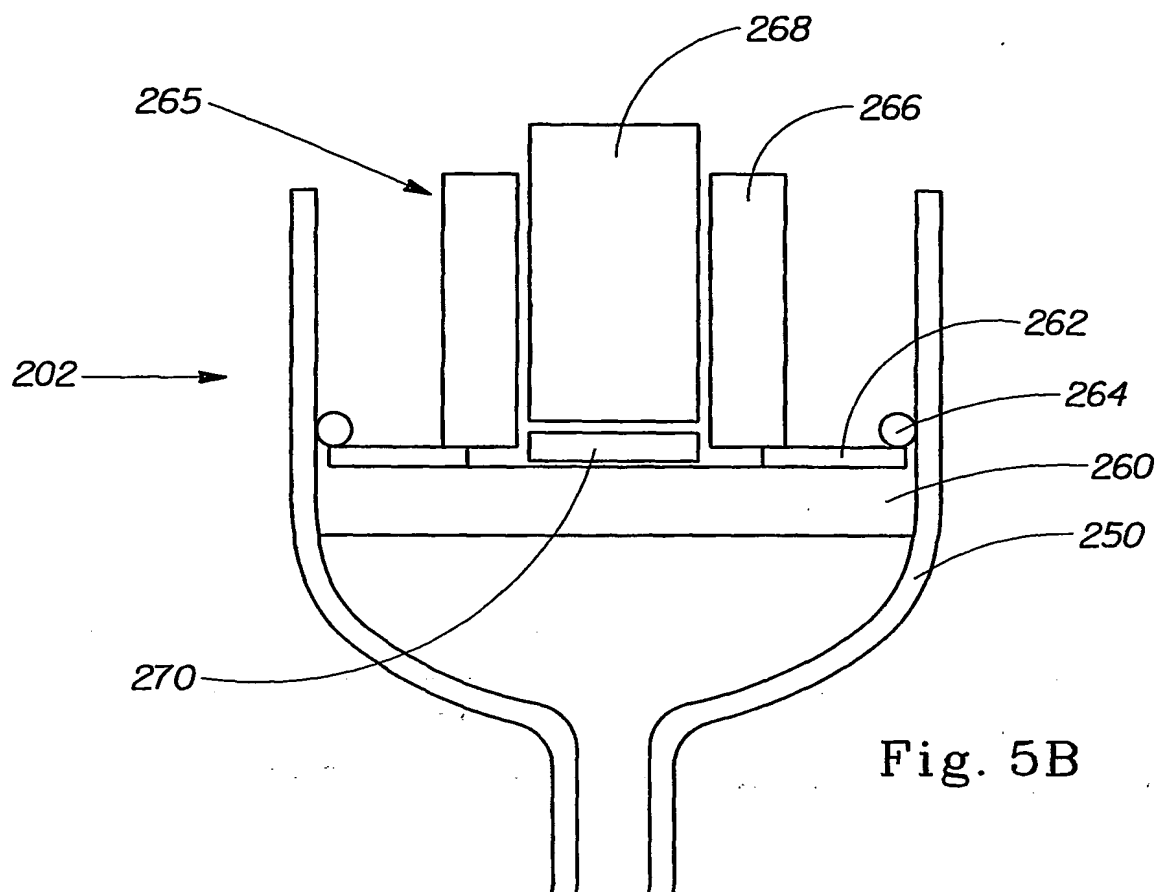


Fig. 5A

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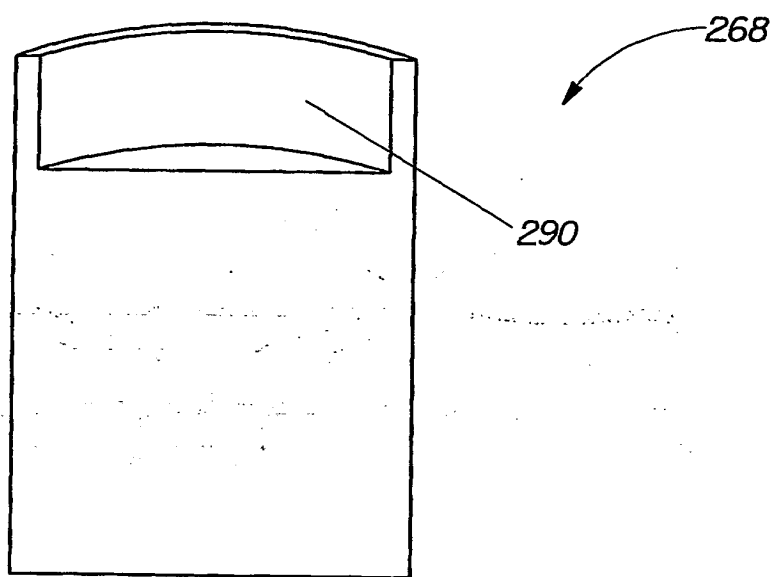


Fig. 5D

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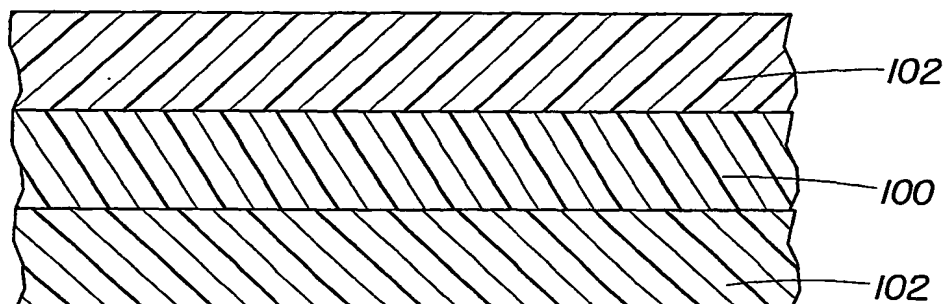


Fig. 6A

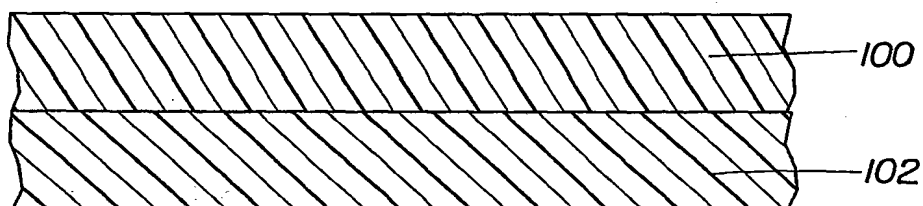


Fig. 6B

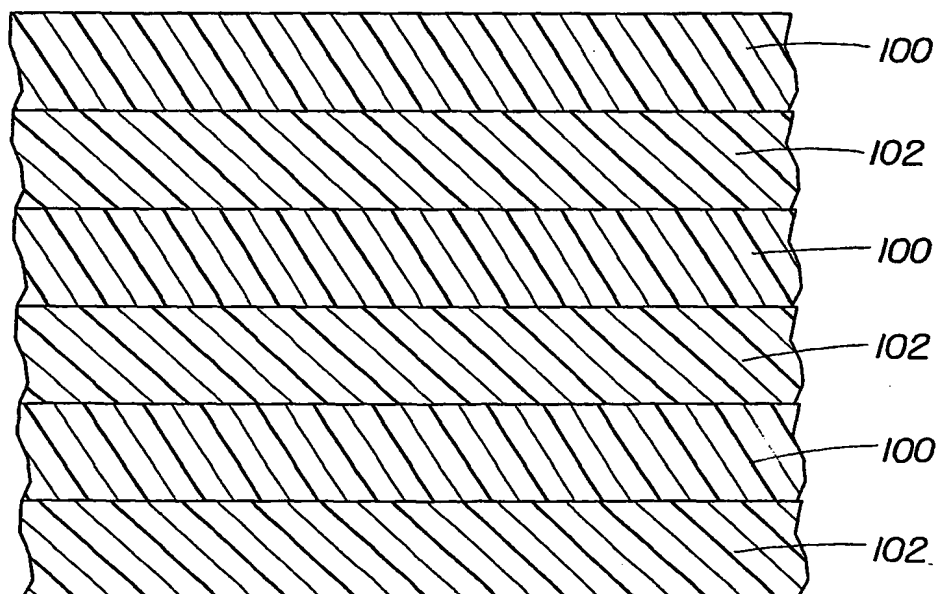


Fig. 6C

# INTERNATIONAL SEARCH REPORT

Inter Application No  
PCT/US 01/09938

## A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 A61F13/15 A61L15/42 A61L15/60

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 A61F A61L

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data, PAJ

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 99 47090 A (PROCTER & GAMBLE) 23 September 1999 (1999-09-23) page 39, line 29 - line 32 figures examples 1-5 claims	1-10
X	WO 99 47184 A (PROCTER & GAMBLE) 23 September 1999 (1999-09-23) cited in the application page 39, line 29 - line 32 page 70, last paragraph -page 72, last paragraph examples 2-5 figures claims	1-10

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

### \* Special categories of cited documents:

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Date of the actual completion of the international search

24 July 2001

Date of mailing of the international search report

01/08/2001

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## INTERNATIONAL SEARCH REPORT

information on patent family members

Intern

Application No

PCT/US 01/09938

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